

JANUARY, 1951

No. 171



Bulletin

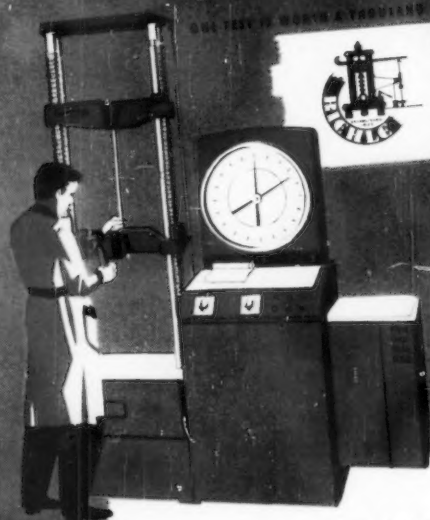
NEWS—Important Accomplishments in 1950; Thermal Insulating Symposium at Spring Meeting; Important New Publications.

PAPERS—Asphalt Paving Mixtures; Surface Waterproofings; Cement Base Paints; Tension Creep-Time Relations; Effective Gage-Length of Tension Bars under Creep; Dielectric Strength at Elevated Temperatures; Emission of Coated Cathodes; X-ray Diffraction Methods Applied to Rubbers.

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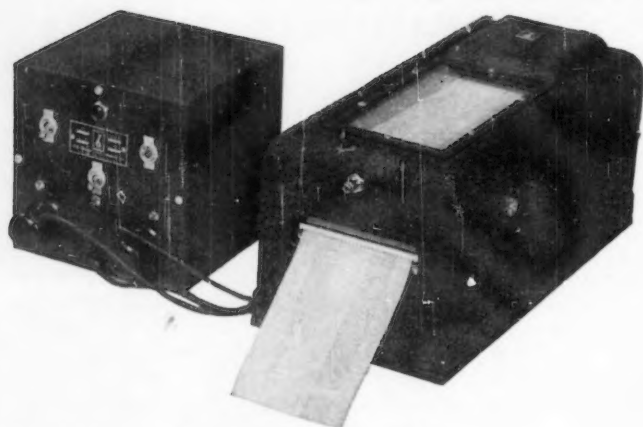
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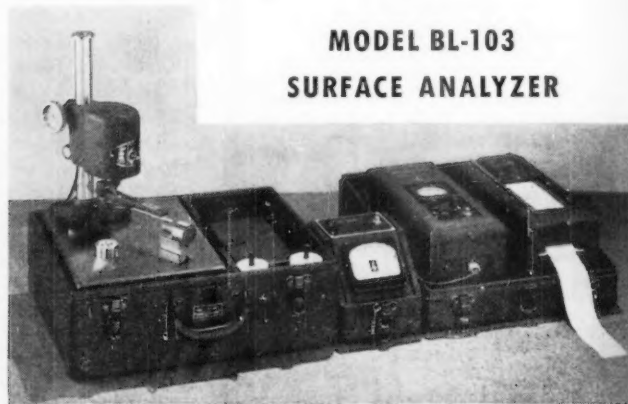
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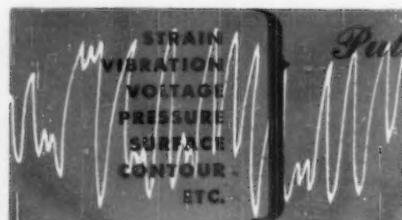
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ASTM BULLETIN

"Promotion of Knowledge of Materials of Engineering, and Standardization of Specifications and Methods of Testing"

TELEPHONE—Rittenhouse 6-5315

R. E. Hess, Editor
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CABLE ADDRESS—TESTING, Philadelphia

Number 171

JANUARY, 1951

Diversity of ASTM Accomplishments in Materials Field During 1950

Important New Standards, Significant Technical Publications, and Expansion Into New Fields Made 1950 a Most Active Year

WITH the great diversity of the Society's work, cutting horizontally across the whole field of materials as it does, there is little possibility of succumbing to the temptation of selecting one major outstanding development for the year and lauding it as an example of what ASTM accomplished. Actually there was no development which one could single out as worthy of top exaltation. Thus even to give a somewhat incomplete notation of important developments we must cover a great variety of fields.

That has been the practice in the past, and probably of necessity will always be so.

In the material on the following pages there are notes on important technical committee accomplishments during 1950. Perhaps not all of the respective leaders would agree on the selections as representing the chief results of intensive work on many fronts, but it appears to the Staff that they are accomplishments of real merit. Actually these notes will provide any members of the Society in a brief reading period with a good cross-section of some of the accomplishments during the year.

New Technical Committees:

In this age of rapid development in the production and use of older materials and the creation of new and improved products, for example, com-

posite materials, ASTM would of necessity, if it were to continue as an important force in bringing about a meeting of the minds of the producer and consumer with respect to the quality and testing of materials, need to continue to expand its activities. The Society's work was extended into several new fields in 1949, and there were important expansions during the past year which should be noted. In 1949 new groups came into existence on such subjects as Chemical-Resistant Mortars (C-3), Acoustical Materials (C-20), and Porcelain Enamel (C-22). During the past year, Committee D-21 on Wax Polishes and Related Material was organized, as was Committee E-13 on Absorption Spectroscopy resulting from a splitting of the work of former Committee E-2 which will now concentrate on Emission Spectroscopy. An agreement was reached to proceed immediately with the development of the Committee on Methods of Atmospheric Sampling and Analysis, to be designated D-22. In recognition of the importance of radioactive materials, Committee E-10 on Radioactive Isotopes was organized to concentrate on the use of these elements in the field of engineering materials, largely their use in analysis and testing. Further announcement of these groups will be made in the ASTM BULLETIN from time to time. (As a matter of fact, one of the im-

portant purposes of the pages of our BULLETIN is to acquaint all of our members with the high lights of committee work.)

New Technical Publications:

The method of insuring that the great and valuable work coming from our technical committees and research men who contribute technical papers hinges on the publication and dissemination of technical books, reports, and papers. During 1950 there were 20 special technical publications issued which covered a great variety of subjects, some of the books being comprehensive in nature, and these were in addition to the numerous other regular or quasi-regular books. For example, the list of STP titles which follows does not include such outstanding special standards books as that covering testing soils or any of the other special compilations of standards. The statement which follows includes a number of references to publications which were sponsored or developed by specific technical committees. Furthermore, the members through a special order blank sent out late in December and through the pages of the BULLETIN have been apprised of the progress in the Society's heavy publication schedule. Without devoting pages to a description of a book, it is virtually impossible to give a complete appraisal. For example,

Are you interested in?: Outstanding ASTM Accomplishments in 1950—p. 5; Spring Meeting on Thermal Insulation and Soils—p. 15; Annual Meeting Symposia—p. 16; Standards Approvals—p. 18; New Publications on Sigma Phase, Quality Control, Identifying Metals, Ultrasonics, Appearance Standards—p. 19; President's Message—p. 25; ASTM Meetings—p. 26; District Activities—p. 27; Technical Committee Notes—p. 29; Mineral Aggregates—p. 32; Asphalt Paving Mixtures—p. 39; Waterproofers and Dampproofers—pp. 48, 53; Creep—pp. 57, 60; Dielectric Strength—p. 63; Cathodes—p. 66; X-ray Diffraction of Rubber—p. 71.

who, without going to considerable length, could describe the value of a Symposium on Plasticity and Creep with its four outstanding papers, or convey adequately the pertinent information and discussion in a Symposium on Turbine Oils. Fortunately all of these publications are readily available and are offered to the members at reduced prices. And, in fact, to all interested the books can be procured at what is considered a very reasonable cost. The Special Technical Publications which came off press during 1950 cover the following subjects:

Metallography in Color
Effects of Low Temperatures on the Properties of Materials
Manual on Fatigue Testing
Testing of Cast Iron with SR-4 Type of Gage
Accelerated Durability Testing of Bituminous Materials
Strength of Wrought Steels at Elevated Temperatures
Data on Corrosion- and Heat-Resistant Steels and Alloys
Evaluation Tests for Stainless Steels
Dynamic Stress Determinations
High Additive Content Oils
Papers on Radiography

Application of Statistics
Use of Pozzolan Materials in Mortars and Concretes
Supplement to the Bibliography and Abstracts on Electrical Contacts
Turbine Oils
Standard Samples for Spectrochemical Analysis
Plasticity and Creep of Metals
Physical Constants of Hydrocarbons
Supplement to the Metal Bibliographical Abstracts
Rapid Methods for Identification of Metals

Technical Meetings:

Fulfilling its long-time responsibility as a forum where the pros and cons of materials problems would be debated and new work covered and evaluated, the Society holds extensive national meetings. The 1950 Annual Meeting was marked by outstanding technical sessions; there were 23 of these not including certain round-table and informal discussions. These, coupled with some 500 meetings of technical committees and an apparatus and photographic exhibit, resulted in the largest registered attendance thus far, exceeding 2100. Some of the publications noted above resulted from this

meeting, and others are to follow. To give some idea of new work—there were over 60 new tentative specifications and tests approved at the meeting.

Many significant committee meetings are held throughout the year. This was true in 1950, and several of the districts sponsored excellent meetings which provided an opportunity for members in the particular localities to greet Society officers and hear various speakers discuss pertinent subjects.

The material which follows, arranged in accordance with the designations of the technical committees, that is, the (A) Ferrous Group, first, followed by the Non-Ferrous (B) Group, then by the Cementitious, Ceramic and Related Materials (C) Group, and last but far from least because of the great extent and variety of their work, the "D" Groups, it is hoped will provide a bird's-eye view of "The ASTM 1950 Panorama of Accomplishments," but like the camera or even the eye when viewing a great vista one must get close to a particular spot if he wishes more detail.

Ferrous and Non Ferrous Metals

Steel and Steel Products—Concrete Reinforcement, Structural Steel and Boiler Plates, Bars

While difficult to select outstanding developments in the work of Committee A-1 on Steel (which has more than 150 specifications and tests in its charge) probably there would be agreement that in 1950 certain work on concrete reinforcing steel, on structural steel, a greatly modified system for specifying steel plates for boilers and pressure vessels, and the completion of specifications for commercial bars would be the major accomplishments. A few notes on these four fields of activity follow.

Concrete Reinforcement Bars:

To Specification A 305 giving minimum recommendations for the deformations of deformed steel bars for concrete reinforcement has been added a series of bar number designations to replace the former size designations, with explanatory notes clarifying the size equivalent of the bar numbers. This action will bring A 305 in conformity with Simplified Practices Recommendation 26 of the Department of Commerce and with practices recommended by producer interests. The changes in A 305 together with the incorporation in Specifications A 15

(now tentative) of revisions involving bend tests have resulted among other developments in covering a new standard high-type bond bar. Further details of these changes and in other reinforcement steels were given in the December, 1950, BULLETIN. Probably no ASTM specifications are more universally used than those covering concrete reinforcement. Subcommittee V of Committee A-1 endeavors constantly to keep them up to date.

Ductility of Structural Steel; Ship Steel:

For decades the minimum elongation (or ductility) requirement of structural steel, a most important property from the standpoint of design and a vital quality in withstanding dynamic stresses for example, has been covered by a sliding scale, a certain percentage of the ultimate strength. Now after intensive studies and debate specific minimum values have been agreed on. This will aid the designer and simplify problems of both the producers and consumers. For example, the minimum elongation (8 in.) in the bridge and building steel specification, one of the first issued by ASTM over a half century ago, has for years read "1,500,000/tensile strength." Now a specific value has been incorporated of 21 per cent, minimum.

Structural Ship Steel.—For some time the Specifications for Structural Steel for Ships (A 131) have been out of date, but because other organization requirements such as the Bureau of Ships, Lloyd's Register of Shipping, and the American Bureau of Shipping covered the material, there was no immediate urgency to revise the standard. However, with demands from consumers and producers that specifications be brought up to date, it has been modernized as a result of studies by a special group.

General and Specific Requirements for Plates for Boilers and Pressure Vessels:

Although the announcement of this important development can be terse, it is nevertheless based on a tremendous amount of work in the Steel Committee's Subcommittee XI on Steel for Boilers and Pressure Vessels. The accomplishment is to set up in one document which includes tolerances, testing requirements, etc., for both carbon and alloy steel, requirements which are common to the some eleven or twelve plate standards with the result that the product specifications are condensed and simplified covering only their respective quality requirements. By this accomplishment Committee A-1 now has two large families of specifications thus covered—structural steels and steel plates. It simplifies publication of the standards, results in more specific cover-

age of matters not heretofore embodied in the product specifications, and the fact that consumers and producers alike are in hearty agreement with the new "system" indicates it is a very constructive accomplishment.

Bar Steels:

For a number of years Subcommittee XV has been writing specifications to cover commercial bars and with the approval by the Society of the Tentative Specifications for Cold-Finished Alloy-Steel Bars (A 330) the major work has been accomplished. There is quite complete coverage of the whole field. Also approved by the Society during the past year are Tentative Methods and Definitions for Mechanical Testing of Steel Bars (A 331). The Steel Committee is hoping eventually to issue a manual on the testing of steel which will comprise chapters covering requirements for specific families of steels. The new tentative A 330 is one of the early steps in this direction.

A special grouping of all the steel bar specifications is in course of publication and will be announced shortly.

Impact Tests of Cast Iron

In the 1949 report of Committee A-3 an appendix reviewed the work of Subcommittee XV on impact test methods applicable to cast iron. As a result of this report the committee drafted the proposed methods of impact testing of cast iron which were published in the 1949 *Proceedings*. In 1950, after a year's study, these proposed methods covering the repeated blow impact test as well as the Izod and Charpy types were issued as tentative (Designation A 327). These new methods should go a long way toward removing misinterpretations of impact testing applied to cast iron.

Corrosion of Iron and Steel—Immersion Tests, Hardware Tests:

The work of Committee A-5 really can be divided into two phases—first, the extensive research investigations being carried on, in large measure atmospheric corrosion tests, and, second, the standardization activities involving specifications and tests. During the year, three developments merit some reference, including new specifications for high-tensile steel telephone wire, and the reports giving the data from total immersion tests and some outdoor tests of hardware and related products.

High-Tensile Telephone and Telegraph Wire:

A number of specifications for various types of wire, wire strand, etc., have been issued, but until early 1950 no requirements had been agreed on for high-

tensile steel telephone and telegraph line wire. The new tentative, A 326, covers two grades of wire with three types of zinc coatings.

Total Immersion Tests:

Although a great amount of time and effort were devoted for many years to immersion tests on copper and non-copper steels, the committee's qualifications as given in the 1950 report can be stated very tersely. The report states that several attempts by various methods have been made to analyze the data statistically to determine if there were a significant difference between copper and noncopper steel. However, when the data are considered from the standpoint of types of steel, both for No. 22 and No. 16 gage sheets, the effect of other elements, and the varying effects of location, the results indicate that any beneficial effect of copper under totally immersed sea-water conditions is so small as not to be of commercial importance. In 1926, a 12-yr. exposure of some low- and high-copper seamless tubing at Portsmouth and at Key West showed no consistent effect of copper under sea-water conditions.

Tests of Hardware:

The atmospheric tests of bare and coated hardware structural shapes and related materials have been very extensive ones and the latest data are

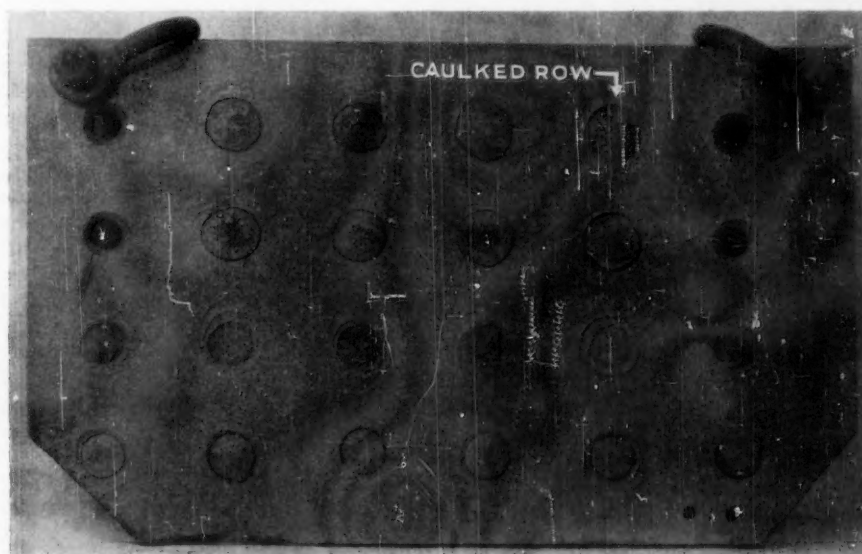
shown in an extensive table in the report. The data from State College and Sandy Hook indicate very slow progress of corrosion of the specimens.

Ferrite Grain Size Standards

Probably every metallurgist is aware of ASTM Standard E 19 providing austenitic grain size standards for Steel, and it is hoped that soon he will be equally aware of the results of long consideration in Committee E-4 which now provide a method for estimating ferrite grain size in steel (E 85). Eight classifications are covered. The standard as published includes a large chart showing the sizes. Grain size in steel seems to be of increasing significance each year. This standard thus will fill a long-felt need.

Electron Microstructure of Steel

Whether the electron microscope could be usefully applied in metallographic work, particularly in studies of some of the fine constituents of steel has been an interesting question which is now largely resolved by the publication of the 1950 Report on Electron Microstructure of Steel. This extensive report, based on a great amount of cooperative work in Committee E-4, with its 47 carefully selected micrographs, shows that this instrument has interesting possibilities and important



This is a replica of a corrosion test specimen in the total immersion tests conducted some years ago to show the effect of sea water on copper- and noncopper-bearing plates and rivets. This particular plate was immersed for some five years at Port Arthur, Texas, and the replica shows the plate after sand blasting. Top row shows low copper steel rivets; second row, high copper steel; third row, low copper puddled iron, and the bottom row, so-called ship quality steel. The chemical composition of rivets and plates is shown in the 1932 ASTM *Proceedings*. Reference is made in the accompanying article to the final report on total immersion tests which indicates that as far as the committee could determine the copper-bearing steel did not show any significant enhanced resistance to the action of sea water over noncopper-bearing steel. The empty rivet holes at the top corners of the plate were left empty so that chains could be attached for suspending the plates.

applications when proper techniques are used. The descriptions of good techniques are one of the real values of the report. This report was described more fully in the September BULLETIN.

Chemical Requirements of Ferro-Alloys

An accomplishment of interest to all those who produce and purchase ferro-alloys is the clarification in the several ASTM standards covering such materials as ferrobore, ferrotitanium, ferromolybdenum, Spiegeleisen, etc., of the chemical requirements which are the basically important properties in the purchase specifications. As a result of a complete review in Committee A-9 there were finally harmonized the views of producers and consumers on how to express clearly and with sufficient accuracy the chemical requirements both as to conformance to the specifications for quality and for commercial negotiations.

Tin and Titanium:

Although no actual standards have resulted as yet from the work of Committee B-2 on Non-Ferrous Metals and Alloys in the field of tin and titanium, it is well to record that this group has accepted the assignment of considering standards for these materials. A number of other committees are concerned with these materials and the committee personnel has been expanded to provide adequate representation.

Corrosion of Magnesium Couples:

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, through its Subcommittee VIII on Galvanic and Electrolytic Corrosion, has an exposure test program consisting of three parts. Part I involves the exposure of disk-type specimens of two types of magnesium alloys coupled galvanically to quite a range of other metals, while Parts II and III will make use of spool-type specimens and plate type, respectively. The disk-type specimens have been assembled and are now on exposure in New York City, Kure Beach, the Canal Zone, and State College. A very considerable amount of work is involved in assembling and preparing the specimens so that this can be considered an important 1950 accomplishment.

Electron Tube Materials:

Because of the importance of the work of Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps functioning under Committee B-4, it is well to note the Proposed Method of Test for Relative

Thermionic Emissive Properties of Materials Used in Electron Tubes prepared by this subcommittee. This is being published to elicit comments and criticism. The test covers a procedure for determining the comparative merits of various radio tube materials based on their effect upon the electrical characteristics of a typical high-vacuum diode (standard diode). Appended to this test are detailed requirements and proposed procedures for the standard diode which is such an important element in evaluating materials for radio tubes and related products.

Copper Water Tubes

For many years, Specification B 88 covering seamless copper water tubes has covered the three types of tubes generally used, but there has been some confusion on which types should be used for underground service, for interior applications, or with soldered fittings. Committee B-5 has added recommendations to the specifications clarifying the application of the three types. The latest recommendations are: type K for underground services and general plumbing purposes; type L for general plumbing purposes, and type M for use with soldered fittings only. Sizes $1\frac{1}{4}$, $1\frac{1}{2}$, and 2 in. in type M are suitable solely for soil, waste, and vent lines, and other interior nonpressure uses.

Mercurous Nitrate Test

Whether the limiting of this widely used test, formerly indicated as suitable for determining the presence of external and internal stresses (stress corrosion as well as season cracking) to apply *only* to internal stresses, is an accomplishment may depend on the viewpoint. Committee B-5 as a result of extensive researches and much discussion narrowed the scope of these Methods B 154. There is also a new note of caution in the method that when it is applied to assemblies and partial assemblies some modifications may be required.

Die-Cast Metals and Alloys:

The outstanding project in the work of Committee B-6 during 1950 was the publication of the extensive data resulting from ten years of exposure of magnesium-base and zinc-base alloys. The report covers the data from five outdoor and three indoor test sites and represents a great amount of interesting and valuable data for the consumers and producers of these materials. The work will go on for total exposure of twenty years.

Pressures used in die casting may have an important effect on the quality of the resulting product, and now that there has been developed a satisfactory method for measuring metal pressures the committee will continue studies of the effect of pressure. In this connection there was published in the May, 1950, BULLETIN a paper covering flow calculations for die casting.

Aluminum Die Forgings and Designations:

Progress to be noted in the work of Committee B-7 on Light Metals and Alloys, Cast and Wrought, includes a new tentative for aluminum alloy die forgings and an extensive editorial revision of numerous aluminum product specifications to incorporate a revised designation system.

Electrodeposited Metallic Coatings:

During the year, Committee B-8 completed exploratory outdoor exposure studies of chromate-treated zinc and cadmium finishes. Plans have been formulated for a more extensive program involving five different treatments.

Although none have yet been published, it is pertinent to note that several recommended practices for plating have been drafted, which when issued should be of widespread interest. This work involves recommendations on the preparation for plating of materials, such as: zinc-base die castings, copper alloys, stainless steel, aluminum, and plastics.

Current Work on Fatigue of Materials:

During 1950 there was issued the first report of a project in Committee E-9 on Fatigue involving a survey of current projects being carried on. This shows that more than 60 laboratories are active on one or more projects, these being noted in the committee's report. With this survey there is given a list of items on which information is needed. The object of this work is to eliminate duplication of effort, if possible, and to note work which might profitably be undertaken.

Chemical Analysis of Metals:

While certainly the outstanding piece of work in the field of chemical analysis in ASTM in 1950 was the completion of the new book of ASTM Methods for Chemical Analysis of Metals, in the development of which the officers and many of the members of Committee E-3 has a vital part, one should not in this case perhaps overlook a few trees for

the forest, which is putting that aphorism somewhat in reverse. The complete book includes much new material, and most previous methods were studied, revised, and expanded to include new and improved procedures on which the committee could agree. For example, the methods applying to lead- and tin-base alloys were either extensively revised or replaced. New methods for nickel-copper and antimony metal are in the book which cover these for the first time. There are new methods for the sampling of non-ferrous metals in cast form, and the volume includes photometric methods for the analysis of aluminum.

Essentially what the Society has to offer the metal chemical analyst is embodied in this book, and yet the committee is continuing to work, studying additional methods, and constantly there is the effort to adopt shorter procedures without sacrifice of the necessary accuracy which is one of the fundamental concepts Committee E-3 keeps in mind and which in the last analysis is probably the major contribution that it makes, among many.

Each member and committee member of the Society received a prospectus describing the 1950 book. Aggregating 486 pages, copies are available in cloth binding at \$6.50 each (\$4.50 to ASTM members); interleaved copies with blank pages for notekeeping are also available at an extra charge.

Effect of Temperature on Metals:

The year 1950 was a notable year for the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals. If nothing else had been accomplished, the technical program which it sponsored at the ASTM Annual Meeting would mark the committee's year as a most productive one. The two-session Symposium on Effect of Sigma Phase on the Properties of Metals, the very interesting and currently pertinent Symposium on Corrosion of Materials at Elevated Temperatures, a round-table session on heavy forgings particularly with relation to gas turbines, and another separate session on effect of temperature with other additional papers attracted the interest of all leaders in this field. All of the technical papers are in course of publication. Some have already been issued, including the notable contribution on "Coal Ash Corrosion of Metals at Elevated Temperatures," by C. T. Evans, Jr., of The Elliott Co.

Reference also should be made to the new Recommended Practice for Conducting Time for Rupture Tests of Metallic Materials (E 85 - 50 T).

Further references to the work of the

committee are included elsewhere in this BULLETIN.

Data on High-Temperature Properties:

One of the important responsibilities of this committee is the compilation of authoritative data on the high-temperature properties of metals, which information is so essential in design and vital for those committees and bodies who are establishing allowable stress values. Through the cooperation of Messrs. R. F. Miller and J. J. Heger a very valuable book was published early in the year providing a

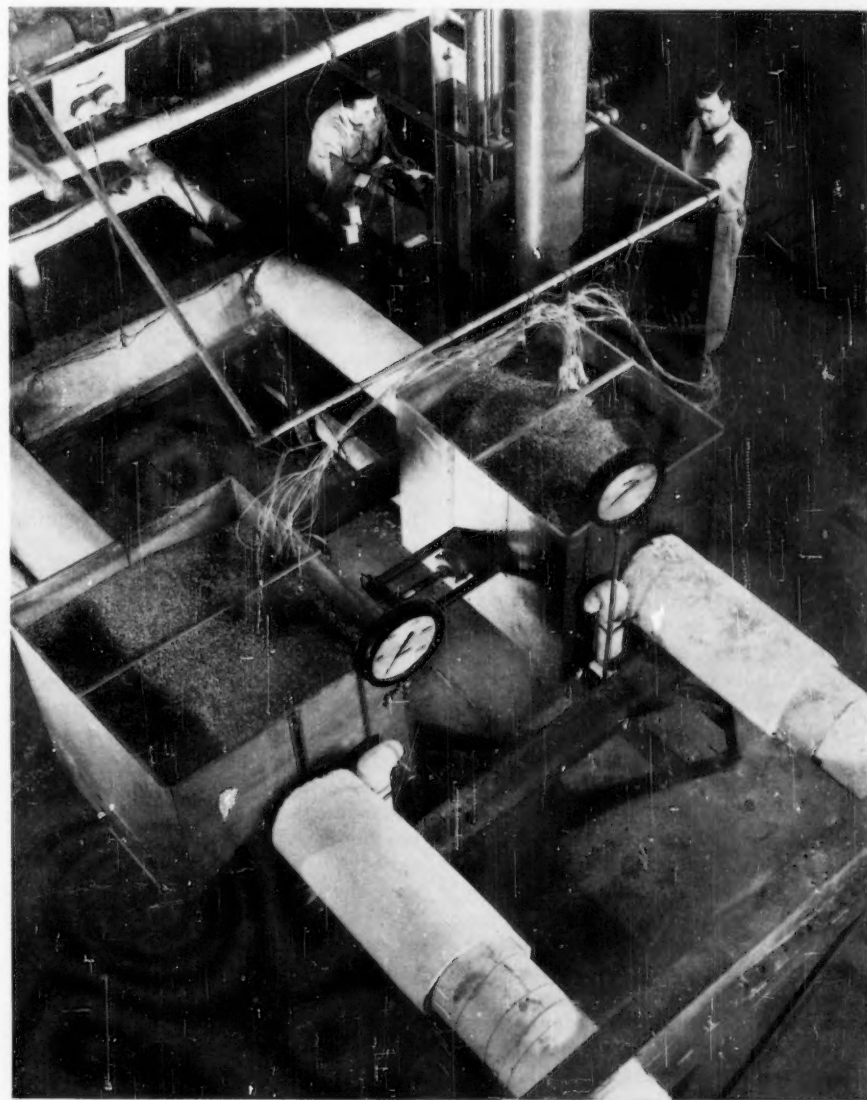
wealth of data on the strength of wrought steels at elevated temperatures. This publication, copies of which are obtainable from ASTM or ASME, is in a sense a "stop-gap" pending the issuance of more comprehensive information which the committee is eliciting from industry. The first part of the data covering steels in the general range of the 18-8 types are being compiled from extensive questionnaires, and a new project will soon proceed covering super alloys and some of the ferritic steels.

Materials Generally Used in Construction

Cement:

Two new tentatives were adopted as a result of work in Committee C-1 on Cement which are of importance and

value to the industry. A Tentative Specification for Air-Entraining Additions for Use in the Manufacture of



"Measuring Temperature Gradients in Insulated Pipe Joints Under Thermal Shock"

An interesting photograph displayed in the General Section of the Seventh ASTM Photographic Exhibit in Atlantic City, by W. M. Pedziwiatr, Crane Co.

Air-Entraining Portland Cement (C 226) will replace and simplify the former procedure whereby each addition was considered individually and accepted by the committee after presentation of sufficient data or after tests sponsored by the committee itself had been carried out. A new Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227) will greatly assist in the determination of this disturbing factor in concrete.

Significant progress was made in research work, particularly in the use of pozzolans. This work will continue.

Oxychloride and Oxysulfate Cements:

This year marked the completion and acceptance by the Society of fourteen tentative methods of testing, representing the first ASTM standards in this field. A list of the materials covered in these new test methods developed in Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements follows: Chemical Analysis of Magnesium Sulfate (C 244), Analysis of Magnesium Chloride (C 245), Testing Magnesia (C 246), Ignition Loss and Active Calcium Oxide (C 247), Slump Test (C 249), Preparation of Specimens (C 251), Linear Contraction (C 252), Linear Change (C 253), Setting Time (C 254), Consistency (C 255), Bulk Density (C 248), Specific Gravity (C 250), Transverse Strength (C 256), Compressive Strength (C 257). Complete titles of these new tentatives appear on page 11, October, 1950, BULLETIN. They are all appearing in the 1950 Supplement to Part 3 of the Book of Standards.

Lime:

Two additional specifications for chemical lime were adopted, namely, Lime for Use in the Production of Grease (C 259) and for Carbide (C 258). This is in the direction of providing chemical lime specifications for each of the several industries in which this material plays an important part.

Refractories:

A tentative method of test for determining the disintegration of fire clay refractories in an atmosphere of carbon monoxide has been approved by the committee. This represents the first result in efforts of the committee to fill a long-felt need for such a method. During the year the latest edition of the widely distributed Manual of ASTM Standards on Refractory Materials was exhausted. It is expected that a new edition of this book, which includes not only the standard specifications and tests, but also much other pertinent

information including numerous industrial surveys of the use of refractories, will be available late in the spring of 1951.

Concrete and Concrete Aggregates:

Significant developments include the adoption of a list of descriptions of various types of rocks. This material is reprinted later in this BULLETIN. This represents the culmination of extended efforts to correlate the many diversified descriptions of aggregate used in the construction field. A complete revision of the specifications for Lightweight Aggregate (C 130) will greatly enhance the value of this specification to the industry in view of the marked increase in the use of lightweight aggregate and in the greater number of sources and types being utilized. There has been much progress in the development of standards for nonair-entraining admixtures, including fly ash.

Gypsum:

Extensive revisions were made in most of the ASTM standards in the gypsum field with the most significant one being a change to the use of compressive rather than tensile strength requirements.

Concrete Pipe:

Tentative revisions dealing with detailed requirements of all of the existing specifications on concrete pipe have been made by Committee C-13, these being the first changes since 1941. In addition, a long-range program has been set up for a complete review of the specifications with revisions which may possibly be based on design requirements. (See page 31)

Glass:

The adoption of a Tentative Method of Sampling and Testing Structural Non-Load-Bearing Cellular Glass Blocks (C 240) provides a useful standard for this popular type construction product. This standard gives brief requirements on sampling and then describes the tests to be applied including density, water absorption, compressive strength, flexural strength, and thermal conductivity. The tests are either delineated in the method or other ASTM applicable procedures are referred to.

Manufactured Masonry Units:

In addition to revisions in the several types of masonry and clay construction units, the committee has now developed a tentative specification for chemical-resistant units—a new field for the committee. This specification includes

a method of test for acid solubility. This past year also marked the completion and acceptance of a complete revision of the old Specifications for Drain Tile (C 4) which have stood since 1924.



A Pulse Receiver Testing Mass Concrete. Described in a paper on dynamic testing by J. R. Leslie, Hydro-Electric Power Commission of Ontario, presented at the 1950 Annual Meeting. This is being published as part of the Symposium on Ultrasonic Testing.

Natural Building Stone:

The adoption of a Tentative Method of Test for Abrasive Resistance of Stone Subjected to Foot Traffic (C 241) provides a useful tool and follows a long period of trial at the National Bureau of Standards and elsewhere. An extensive exposure test wall consisting of practically every known type of natural building stone used in construction has been completed and is located on the grounds of the National Bureau of Standards. This wall was not directly a project of the committee but the committee will have joint sponsorship with the Bureau in recording of data and history.

Structural Sandwich Constructions:

Four proposed tentative methods have now been agreed upon by Committee C-19 representing the first ASTM standards developed for this new type of construction. Two of these methods are used for determining water absorption and specific gravity of core materials and two are for determining compressive and bending flat-wise strength tests on sandwich constructions. These four methods will

be presented to the Society's Committee on Standards in the near future.

Ceramic Whiteware:

The adoption by the Society of 26 definitions covering terms in the field of ceramic whitewares comprises the initial efforts of the new Committee C-21 organized in 1948. Agreement on description of terms of this type has been a very difficult one, and much credit must be given to the group which

has developed these definitions. Among some of the terms covered in this new tentative definition (C 242) are the following: Ceramic whiteware (a fired ware consisting of a glazed or unglazed ceramic body which is commonly white. This is a generic term comprising chinaware, porcelains, earthenwares, etc.); china, porcelain, earthenware, slip coating, technically vitreous, highly porous, frit, vitrification, etc.

Miscellaneous Materials, Petroleum Products, Paints, Rubber, Textiles, Etc; and Some Construction Materials

Paints and Related Materials—Solvents, Pigments, Exposure Forms:

Committee D-1 on Paint, Varnish, Lacquer, and Related Products has a very large number of standard specifications and tests in its charge. Keeping these up to date, perfecting new methods and specifications—because there is always much progress being made in this field—and sponsoring the needed research work, all make for great activity. During 1950, the committee's numerous accomplishments were highlighted by new tests for volatile hydrocarbon solvents, new and revised analytical methods for pigments, and the development of paint panel record forms for recording exposure data.

Solvents:

The subcommittee on volatile solvents for organic protective coatings completed three important new methods covering Heptane Number (D 1132), Kauri-Butanol Value (D 1133), and Diluting Power (D 1134). The first two items are for determining the relative solvent power of the materials; the first, of high solvency materials in the presence of certain resins; the second, for materials used in paint and lacquer formulas.

Chemical Analysis of Pigments:

The chemical analysis of pigments engages the active attention of Subcommittee VIII. During the year it completed important revisions and perfected several new methods. The current methods, D 50, covering yellow, orange, red, and brown pigments were revised and the new procedures were issued covering the following: Zinc Dust (D 521); Blue Pigments (D 1135); and Chromium Pigments (D 126), the latter being essentially an extensive revision.

Paint Panel Record Forms:

With hundreds of paint exposure

programs under way, there has been evident a need for some standardized record form, and this will now be available with the single and multipanel forms that the Society is publishing as developed by Committee D-1 in cooperation with the Federation of Paint and Varnish Production Clubs. The multipanel type, essentially for field observation, provides for 20 different observations of 20 different panels. These data then can be transferred to the single panel form which will cover 15 or more types of failure, over a period of five years. Further information will be published concerning these forms which it is hoped will be available late in January. With the forms there is a copy of the Tentative Description D 1150.

Petroleum Products and Lubricants:

The term "Committee D-2 on Petroleum Products and Lubricants" is synonymous with a tremendous program of work and activity. The annual compilation of standards sponsored by this group, which also includes related information, now comprises over 780 pages and even this does not include all of the standards, because the widely distributed ASTM Manual of Engine Test Methods for Rating Fuels includes standard methods not in the D-2 compilation, and the prospective new book on measurement and sampling will have others. Seventeen new tentatives alone came from the D-2 work during 1950, with numerous revisions of standards and important other achievements. Some are noted here.

Manual on Measurement and Sampling of Petroleum and Petroleum Products:

Culminating some ten years of work, with publication imminent, although temporarily delayed, is the new Manual on Measurement and Sampling of Petroleum and Petroleum Products

which makes available in convenient form what are believed to be the best and most accurate methods for measuring the volume of petroleum and obtaining representative samples. This publication, profusely illustrated, will be useful to the producer in the field, to those concerned with transportation, to the refiner, and all those who must arrange for the sampling and measurement of petroleum and its products. A detailed announcement of this publication was sent to the members in December.

Important New Petroleum Tentatives:

New ASTM standards are always of special significance to certain groups. In a particular industry they may be of great service to some segments and of only normal interest to others, hence the designation as outstanding of new methods developed by Committee D-2 needs qualification; however, in any list of significant developments one must include the new tentatives covering Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases (D 1090); the Apparent Viscosity of Lubricating Greases (D 1092); and Methods for Boiling Point Range of Polymerization-Grade Butadiene (D 1088) and the Carbonyl Content of Butadiene (D 1089).

Proposed Methods:

The Petroleum Committee frequently publishes for a year or more to elicit comments and criticisms various proposed tests and important procedures. In this category in the 1950 ASTM D-2 compilation are methods for reduced pressure distillation, for hydrocarbon analysis of Diesel fuels, and new tests for refractive index and for density of hydrocarbon liquids. There is also proposed a test for evaluating the pour stability characteristics of winter grade motor oils. The latter is based on very extensive research work over several years, with several laboratories cooperating. This research and summary of results is described on page 665 of the D-2 book. This work is one of many examples where extremely extensive field and laboratory test work must be

done to establish the validity of a proposed standard test.

Gaseous Fuels:

A great amount of research work, much of it of a cooperative nature, carried out in Committee D-3 on Gaseous Fuels has resulted during the year in three important analytical methods. One of these described elsewhere in this BULLETIN (see article on December Standards) covers the Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature (D 1142). The other two cover the Analysis of Natural Gases by the Volumetric-Chemical Method (D 1136) and by the Mass Spectrometer (D 1137). The first method is to be used when complete composition is *not* required. The data obtained by this method are usually sufficient for the calculation of heating value and specific gravity, and are quite satisfactory for the calculation of combustion characteristics. The method combines absorption and combustion methods. Acid gases, oxygen, and unsaturated hydrocarbons are absorbed; saturated hydrocarbons are burned with excess oxygen over hot platinum; and the contraction, carbon dioxide produced, and oxygen consumed are all measured. The second method, using the mass spectrometer, will give the complete chemical composition of natural gases and gaseous mixtures through the C_4 hydrocarbons, including information concerning vapors of heavier hydrocarbons which may be present. The molecular species making up a gaseous mixture are ionized and dissociated by electron bombardment, the resulting positive ions of different masses are accelerated in an electric field and are separated magnetically, and the abundance of each mass present is recorded. The mixture spectrogram thus obtained is solved against the mass spectra of each of the pure molecular species constituting the mixture.

Road and Paving Materials:

New ASTM tentatives which are significant revolve around bituminous mixtures. Three tentative tests cover Bitumen Content of Paving Mixtures (D 1097), Compressive Strength of Bituminous Mixtures (D 1074), and Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075). One new tentative specification in this field is that for Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073).

The subject of triaxial testing has received much attention and Committee D-4 participated in a symposium which was presented at the Pacific Area Meet-

ing of the Society, the papers of which will be included in a publication jointly with those on soils.

Paper:

The adoption of a specification for filter paper for use in chemical analysis marked the first specification which Committee D-6 has completed. Heretofore the work in the committee has been concentrated on the development of test methods. New tentative methods accepted cover the static bending of corrugated board and the determination of water-soluble solvents in paper and paperboard.

Wood:

Ten new tentatives, nine being chemical methods, mark a considerable as well as initial development in the activities of Committee D-7 in the field of chemical analysis of wood. These methods cover Ash (D 1102), Water Solubility (D 1110), Ether-Solubility (D 1108), Alcohol-Benzene Solubility (D 1107), One Per Cent Caustic Soda Solubility (D 1109), Holocellulose (D 1104), Lignin (D 1106), and Alpha-cellulose (D 1103). The other new tentative, covering the Testing of the Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101) provides a very useful procedure for measuring quality. A secondary method has been added to the Standard Methods of Testing Small Clear Specimens of Timber (D 1043) intended for use in evaluating the properties of wood when only relatively small trees, generally less than 12 in. in diameter, are available to provide the test specimens.

Bituminous Waterproofing and Roofing:

The first group of definitions in this field were approved during the past year, this accomplishment representing many years of effort in Committee D-8. Some of the terms covered are as follows: asphalt mastie, bituminous grout, built-up roofing, cap sheet, exposure, membrane, mineral granules, pitch, rag roofing felt, selvage, etc.

Electrical Insulating Materials—Condenser Paper and Oils:

Probably the two major accomplishments among the several projects completed by Committee D-9 during 1950 were important additions to the Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202) and a complete revision, with considerable new added material, of the Methods of Testing Electrical In-

sulating Oils (D 117). Added to Methods D 202 are the procedures covering the determination of the extent of water and alcohol-soluble matters. More sensitive methods for measuring heat stability have been included and tests covering Fiber Analysis of Paper (D 1030) and Water-Soluble Sulfates (D 1099) are referred to. Extending the policy in Committee D-9 of including statements of the significance of various tests, several have been incorporated in the methods relating, for example, to thickness, bursting strength, folding endurance, etc.

The use of tremendous volumes of electrical insulating oils makes the methods of evaluating these materials very important, hence it is notable that the ASTM Methods D 117 applying to these oils have been completely revised with several new test methods included. These methods cover oils used in cables, transformers, circuit breakers, and other equipment, and the methods apply to numerous properties such as color, dielectric strength, gas content, power factor, resistivity, viscosity, etc.

Shipping Containers:

During the past year Committee D-10 made considerable progress in the collection of performance standards data. Its work is definitely trending toward performance standards as distinct from the efforts of the committee during the past few years in developing laboratory and related testing procedures, for example, drop tests, incline impact test, water resistance, and others.

Expansion of the scope of Committee D-10 is significant in that it now will include the development of standards in the entire field of packaging, including packages, packing, shipping containers, and pallets. A further achievement has been the completion of a glossary of terms relating to interior packaging which will be included in the Tentative Definition of Terms Relating to Shipping Containers (D 996) as well as a Method of Testing Large Shipping Cases and Crates (D 1083). It is indicated that the method is suitable for testing large containers, and as well the ability of the contents and fastenings to endure normal handling. Five methods are covered including a drop test, superimposed load test, grabhook test, sling test, and railroad handling test.

Rubber and Rubber-Like Materials:

Committee D-11 has prepared many methods for determining the chemical and physical properties of rubber products and has issued numerous specifications. Among the methods, those for Chemical Analysis of Rubber Products (D 297) are very important; a major 1950 accomplishment has been the complete rewriting of the methods. Additional definitions have been given and a more usable form has been embodied; and of considerable significance along standardization lines is the coordinating of procedures with those of the Federal Specifications Board. These methods are arranged into three separate procedures:

A. Complete Procedure. B. Direct Determination of Rubber Hydrocarbon. C. Copper and Manganese Determination in Crude Rubber. Method A is intended for general use in the determination of some or all of the major constituents of a rubber compound. It is designed primarily for use with natural rubber compounds, but many parts of it may be applied to synthetic elastomer compounds or mixtures of these with natural rubber compounds. Method B is a procedure for the direct determination of natural rubber hydrocarbon by quantitative oxidation with chromic acid and measurement of the acetic acid thereby produced. Method C includes specialized procedures for the determination of copper and manganese in crude natural rubber only.

Important revisions of the methods of Testing Rubber Insulated Wire and Cable (D 470) were made. They cover the determination of ozone concentration by either the present chemical analysis method or by direct measurement with an ozonometer. The latter has the advantage of permitting continuous readings of the ozone concentration during test.

Soaps and Other Detergents; Metal Cleaning:

Two significant new specifications covering soap where comparatively low-temperature washing solutions are indicated, were issued through the work of Committee D-12, these specifications covering Solid Soap (D 1112) and Chip or Granular Soap (D 1117). These soaps are intended primarily for use in cleaning wool and other fabrics, finished surfaces and other articles. In its report the committee published two proposed methods to elicit comments, one a Test for pH of Aqueous Solutions of Soaps and Detergents, and the other a Test for Foaming Properties of Surface-Active Agents.

Metal Cleaning Bibliography.—Through the efforts of J. C. Harris,

former secretary and now chairman of Committee D-12, there was issued in 1949 a very extensive book providing bibliographical abstracts on metal cleaning. In 1950 an extensive supplement to this book was issued under the auspices of Committee D-12. There are many hundreds of references and abstracts, thus making available in compact form a great wealth of information which otherwise is scattered widely throughout the technical literature. Copies are available from ASTM Headquarters.

Textile Materials:

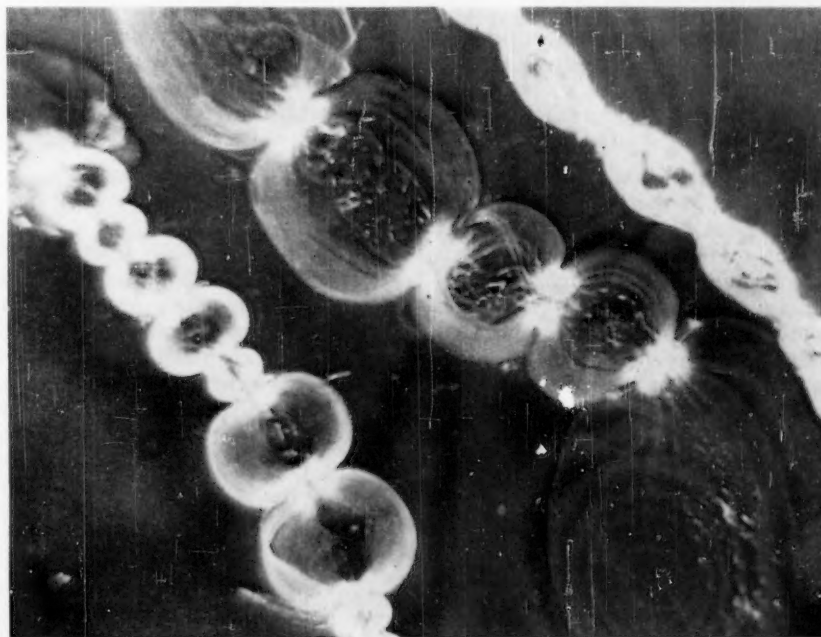
Some of the standardization projects completed by Committee D-13 are of special interest to certain segments in the textile field, although the activities of this committee cover rather completely all of the principal textile materials. A major development was the completion of extensive revisions in the Methods for Resistance of Textile Fabrics to Water (D 583). Some of the procedures embodied are based on methods developed by the American Association of Textile Chemists and Colorists, and in others there was close cooperation with the U. S. Army. These methods of test are intended for the determination of resistance of textile fabrics to (a) external (surface) wetting, (b) internal wetting (absorption), and (c) penetration by water. They are applicable to all fabrics, both treated

and untreated, regardless of fiber composition.

The first set of methods applicable to materials called "bonded fabrics" were approved, carrying the designation D 1117. Bonded fabrics are described in the "Scope" clause, which reads:

These methods of test are applicable to fabrics which are neither woven, knitted or spun, but built up by the interlocking of fibers by a chemical bonding material, by mechanical work, chemical action, moisture, and heat. Bonded fabrics may be produced by any one or a combination of these factors. It may consist of cotton, cotton mixed with other fibers, or fibers other than cotton. It is defined as a structure consisting of a web or mat of fibers held together with a bonding material.

Some idea of the range of activities of the committee can be had from a list of other important developments during the year. These include a new Tentative Method of Test for Snag Resistance of Hosiery (D 1115); a procedure for determining the Resistance of Pile Floor Coverings to Insect Pest Damage (D 1116) which makes use of the black carpet beetle as the testing agent; also tests for Vegetable Matter in Scoured Wool (D 1113)—in this case vegetable matter refers to impurities of plant origin, such as seeds, burs, twigs, leaves, and grasses; and the new Tentative Specifications for Mechanical Sheet Felt (D 1114), these requirements applying to a felt which is commonly referred to as wool felt.



"Cotton Fibers Swelled in Cupri-Ethylene Diamine Phase"

Third prize-winning photomicrograph in the Seventh ASTM Photographic Exhibit by John M. Facq, Bates Mfg. Co. Original magnification 400 X—final magnification 1200 X.

Adhesives:

Continued emphasis by Committee D-14 on the development of test methods resulted in the approval by the committee during the year of a tentative method for flexural strength and publication by the Society of the Tentative Method of Test for Consistency (D 1084). Considerable progress has been made in the difficult field of writing specifications for adhesives on a performance basis. The pattern has been to formulate specifications for specific uses, such as, acoustical tile, book binding, and general purpose.

Engine Antifreezes:

Although Committee D-15 actually has not been in operation much more than a year, it has carried out some very intensive work including a number of cooperative studies. There has been evident a real need for some research and standardization activities so that the several new standards issued in 1950 will fill long-felt needs. In addition to Specifications for a Field Tester for Engine Antifreezes of a Hydrometer-Thermometer type (D 1124), five test methods were completed, four of which apply to concentrated engine antifreezes, the properties evaluated being Reserve Alkalinity (D 1121), Specific Gravity (D 1122), Water (D 1123), and Ash (D 1124). Of much importance also is the new Method of Test for Boiling Point of Engine Antifreezes (D 1120). These tentative standards were published in the 1950 Report of Committee D-15 and will appear in the 1950 Supplement to Part 5 of the Book of Standards.

Soils:

It is recognized that the development of standards in the field of soils engineering must be preceded by a great amount of research and development of techniques. Since its complete reorganization, Committee D-18 has been sponsoring extensive research in several phases. Accomplishment denoting progress in the field has been shown by the presentation of an extensive Symposium on Triaxial Testing of Soils at the 1950 Annual Meeting and a second Symposium on Identification and Classification of Soils. A greatly expanded and revised edition of the "Procedures for Testing Soils" was published during the year. This includes not only the 17 methods which have been developed by the committee, but many more proposed procedures that are in use throughout the country. Some of these are being studied by the committee, but all have been edited and published in

the style followed in publishing standards. One of the methods, as noted elsewhere in this BULLETIN in the article on "Standards Actions," has already received Society approval, this covering load settlement of individual piles.

Industrial Water:

1950 was a productive year for Committee D-19, with several new test methods issued and other work completed. Included in the new standards are Methods for Determining Sodium and Potassium in Industrial Water (D 1127) and a procedure for Testing Electrical Conductivity (D 1125). The latter is applicable for the detection of ionized impurities dissolved in, condensed steam, the approximate determination of dissolved electrolytes in natural, treated, and industrial waters, etc. It does not attempt to cover research techniques or apparatus. Almost everyone has talked about "hard" or "soft" water, and while others may have done something about it, Committee D-19 definitely has, through its new Test for Hardness in Industrial Water (D 1126). In this method, which is a volumetric one, a standardized soap solution is used.

Another method which should be noted as a significant advance during 1950 is the Method of Identification of Types of Microorganisms in Industrial Water (D 1128). This tentative uses microscopic examination to identify bacterial cells, mold filaments, and inert material. It includes a condensed key for identification of the microorganisms.

Another constructive project in Committee D-19 was the compilation in one document of various standard definitions of terms which carries the designation D 1129.

Plastics:

The Society's Committee on Plastics always has an active program of research and standardization. Many current projects are noted in the 1950 D-20 Report but reference should be made here to two significant standards completed during the year. While the new Recommended Practice for Injection Molding of Specimens of Thermoplastic Materials (D 1130) covers only the general principles this important document will establish uniformity in preparing specimens. Its use will result in uniform methods of describing the various component parts of the molding operation and also set up uniform methods of reporting these conditions.

Also to be noted is the extensively revised Test for Luminous Reflectance,

and Transmission Characteristics and Color of Plastic Materials (D 791). Following revision this was adopted as standard. While it applies to substantially nonfluorescent plastics, other materials may be tested by it. It is based upon the Hardy-type spectrophotometer.

Methods of Testing—Brinell Hardness; Verifying Extensometers; Thermometers:

Probably no test is more widely applied to metals than the hardness test and consequently the ASTM standards for hardness testing of metallic materials are most important. This year Committee E-1 developed revisions in the Brinell Hardness Test (E 10) which now provides for use of the 1500-kg. load for metals of intermediate hardness, in addition to the 500 and 3000-kg. loads. Another important change covered limits for use of various balls, recommending a steel ball on material having a Brinell hardness number not over 450, a Hultgren ball for not over 500, and a carbide ball not over 630. Suggested loads in the revised methods are based on the following hardness limits: 3000-kg. load between 160 to 600 B.H.N., 1500-kg. load between 80 and 300 B.H.N., and 500-kg. load between 26 and 100 B.H.N.

Another important responsibility of this committee is the establishment of standard methods of verifying testing machines and such equipment. A new Method of Verification and Classification of Extensometers (E 83) was approved. These devices are widely used for measuring strain and may be either of the indicating or recording type.

The work of Committee E-1 has been outstanding in clarifying and establishing standard requirements for a wide range of thermometers used in various ASTM standards. The committee also has issued an important method of Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77). In 1950 specifications were completed for a set of 18 precision fractionally graduated test thermometers; also new specifications for Butadiene Boiling Point Range Thermometer and revised Stability Nitrocellulose Test Thermometer.

Emission Spectroscopy:

While there is no published material to indicate the intensive work carried out during 1950 by Committee E-2, nevertheless this group has a large number of suggested methods for emission spectrochemical analysis virtually completed, and these methods will be issued in 1951, at which time further announce-

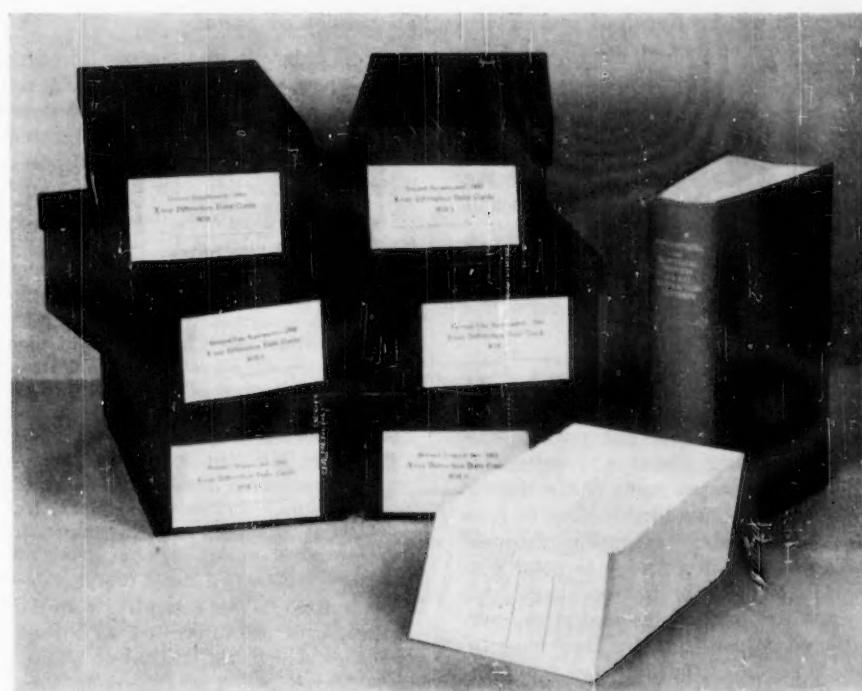
ment will be made. Members of the committee have been devoting much time and effort to the work, and because of the steadily growing interest in this field the results will be of widespread interest and service.

Non-Destructive Testing Symposium

In course of publication (guesstimated date, February, 1950) are the several interesting papers comprising the Symposium on Non-Destructive Testing held during the Annual Meeting under the auspices of Committee E-7. The papers relate to the use of radiography, ultrasonics, magnetic particle testing, and liquid penetrant inspection. The publication is intended to evaluate the practical applications of various non-destructive tests, and an advance review of the publication indicates widespread and increasing use of the various methods. In a sense this symposium might be considered a down-to-earth report from the technical and engineering fraternity to management.

Radiographic Testing

The drafting of a proposed recommended practice for radiographic testing, credit for which should go largely to Dr. H. E. Seemann, chairman of this subcommittee in Committee E-7 on Non-Destructive Testing, merits mention at least, in any review of 1950 work. The method has not been approved yet in Committee E-7 and is simply being distributed for comment and criticism, but the task was such an arduous one that it was quite long delayed. There is real need for a standard practice to replace the former ASTM methods which were dropped because of obsolescence several years ago.



The Three Sections of the X-ray Diffraction Data Cards File Totalling over 4000 Cards. Widely used in work of all kinds involving the identification and analysis of many materials. The Index is included with the set. Cost for the complete file, \$405; in Keysort, \$555.

Card Files of X-ray Diffraction Patterns

The issuance of the extensively revised card files of both plain and Keysort cards of X-ray diffraction data to be used in X-ray testing and research involving the identification of crystalline materials is one of the major jobs completed during the year. In this work ASTM had a leading part in cooperation with other societies. These card files are virtually essential in the identification and use of X-ray

patterns. And with the X-ray widely used in analyzing and identifying crystalline materials almost horizontally across industry, these card files become one of the most important adjuncts of the X-ray diffraction machine. Even though the purchase price of a complete set of these cards may run about \$400, their value in X-ray work is incalculable, because no single company or group of companies would be in a position to compile the material and issue it the way the committee has done, except by Herculean efforts.

1951 Spring Meeting to Feature Thermal Insulating Materials Symposium

Session on Soils Also to Be Held During Week of March 5

IN RECENT BULLETINS, it has been announced that the ASTM Spring Meeting would be held in Cincinnati this coming year during the first week in March. Present plans call for an afternoon session and for a talk in the evening to be delivered by some speaker of prominence in a field of engineering materials. The highlight of the afternoon session will be a symposium on thermal insulating materials which will include several papers by men eminent in their particular specialty.

Charles F. Gilbo, Armstrong Cork

Co., will present a paper "Experiments with a Guarded Hot Plate Thermal Conductivity Set" in which he will describe equipment and limits which must be met in order to operate this apparatus successfully. The effect of ambient temperature on guarded hot plate results, the effect of thermal unbalance between the heater and guard, and the correlation of errors in results with equipment faults will be discussed in the paper by Mr. Gilbo.

In "The Measurement and Significance of Specific Heat of Thermal Insu-

lating Materials" by Norman H. Spear John B. Pierce Foundation, a discussion will be presented on the basic significance of the specific heat property in the thermal performance and response characteristics of insulating materials. Classical specific heat measurements with reference to individual materials will be considered. By a number of illustrative examples, primary problems in the practical field will also be discussed. These include analysis of the effect of specific heat on the performance of building structures subject to different diurnal and seasonal temperature cycles, a consideration of pipe insulation practice, and a review of recent work in engineering properties of protective clothing. A tried and tested method of specific heat measurement on insulating

materials will be discussed and details will be presented as an appendix to the paper.

Based upon experiments which have continued for the past three years at the Pennsylvania State College under the sponsorship of the U. S. Housing and Home Finance Agency and the Office of Technical Services of the U. S. Department of Commerce, Frank A. Joy has written a paper which will be part of the symposium. His "Basic Concepts of Water Vapor Migration and Their Application to Frame Walls" should throw more light on a technical problem in the building industry which has become quite significant in recent years. Mr. Joy will review some of the theory which has been used in the past to predict water vapor migration through walls and he will attempt to point out shortcomings in the theory where they exist. Mr. Joy and his associates have gathered data with which they hope to clear up some of the confusion and misconceptions which at present are connected with some of these basic conceptions.

"The Appropriate Use of Different Methods in the Measurement of Surface Emissivity and Emittance" by L. P. Herrington, John B. Pierce Foundation, is an analysis of methods as applied to fabrications. In his paper, primary emphasis will be given to factors of accuracy and convenience involved in the general extension of these methods to a wide range of materials with a discussion of the practical implications of the measurement in estimating the thermal performance of material assemblies.

It is also expected that a paper by H. E. Robinson, National Bureau of Standards, will be presented concerning round-robin type thermal conductivity test.

Discussion on Soils:

In addition to the Spring Meeting Symposium on Thermal Insulating Materials, other groups may sponsor technical discussions and Committee D-18 on Soils for Engineering Purposes definitely is arranging for the presentation of three technical papers on subjects of great interest to all those who are working in this field. These papers are as follows:

Identification of Clays by Staining Tests—R. C. Mielenz and M. E. King
An Analysis of the Effect of Particle Interlocking on the Strength of Cohesionless Soil—B. K. Hough
De-aired, Extruded Soil Specimens for Research and Evaluation of Test Procedures—L. Hudson Matlock, Jr., Carl W. Fenske, and Raymond W. Dawson

Committee D-18 is extending a cordial invitation to all interested to attend its session which will be held on Thursday, March 8. These papers were procured for the committee by Prof. R. F. Dawson, University of Texas, who is chairman of Committee D-18's subgroup on special papers.

Committee Week

THROUGHOUT the week beginning March 5 there will be held at the Netherland Plaza Hotel in Cincinnati hundreds of meetings of ASTM technical committees, which groups will take advantage of the annual Committee Week to get together and act on their many standardization and research projects. There is a somewhat different pattern of committees meeting each year, although some of the technical groups always participate. A list of the main groups which have thus far signified their intention of

meeting in Cincinnati in 1951 is as follows:

A-3 on Cast Iron
A-5 on Corrosion of Iron and Steel
A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys
B-2 on Non-Ferrous Metals and Alloys
B-6 on Die-Cast Metals and Alloys
B-7 on Light Metals and Alloys, Cast and Wrought
B-8 on Electrodeposited Metallic Coatings
C-1 on Cement
C-3 on Chemical-Resistant Mortars
C-7 on Lime
C-8 on Refractories
C-9 on Concrete and Concrete Aggregates
C-11 on Gypsum
C-12 on Mortars for Unit Masonry
C-15 on Manufactured Masonry Units
C-16 on Thermal Insulating Materials
C-17 on Asbestos-Cement Products
C-22 on Porcelain Enamel
D-3 on Gaseous Fuels
D-4 on Road and Paving Materials
D-5 on Coal and Coke
D-8 on Bituminous Waterproofing and Roofing Materials
D-17 on Naval Stores
D-18 on Soils for Engineering Purposes
D-19 on Industrial Water
E-1 on Methods of Testing
E-9 on Fatigue

Many of these groups will have subcommittee and section meetings, and while the secretary of each committee will be advising his members on the time they will meet, also there will be mailed out to all the members from ASTM Headquarters about January 20 a combined schedule of all the meetings. *A hotel return form for sleeping accommodations will be included. No problems in this respect are anticipated this year.*

In preparing the master schedule, an earnest effort is made to keep the number of conflicts to a minimum. A basic purpose of Committee Week is to permit a large number of the men who serve on different technical committees to attend meetings of these frequently diverse groups in a concentrated period.

Significant Technical Symposia Planned for 1951 Annual Meeting

IN THE December BULLETIN there were thumbnail sketches of the several symposia which are to be held at the Annual Meeting in Atlantic City, June 18 to 22, 1951. Since then, authors of papers that are part of these symposia have supplied additional information concerning their topics. Although all summaries have not been

received, the paragraphs below should give some indication of what is in store.

Flame Photometry Symposium

A rapid method for determining the calcium content of oil has been devised using the flame spectrophotometer. The accuracy of the method to be described is comparable with that of ASTM's Methods of Chemical Analysis for Metals in Lubricating Oils (D 811) above 0.1

per cent, and at lower concentrations the flame spectrophotometer is even more accurate. A further advantage ascribed is that three samples can be run in one hour.

Also V. W. Meloche's paper will constitute a brief review of the historical development of flame photometry stressing limitations and advantages. A set of experiments designed to uncover weak points in the methods by correlations of atomizer geometry and other variables, and possibilities of broadening the field of application will be discussed.

"The Effect of Organic Solvents on the Flame Photometric Emission of Certain Elements," by Curtis, Knauer, and Hunter, will explore the possibility that

nonaqueous materials may exert an influence upon the emission characteristics of metals. A variety of compounds have been studied and the data indicate that the solvent influences extent of emission and that, in some cases, an enhanced emission may be used to increase the sensitivity of analyses.

Eggertsen, Wyld, and Lykker, show that the interferences of various chemicals in the determination of sodium by flame photometry are shown to be of the same general character and that the addition of an excess of one salt to both sample and standard can be used to buffer out the interference of all the others. Also the authors discuss the atomizer's and spray chamber's relation to interference and experiments in which a modified separate assembly was used. A possible mechanism for one class of interference is suggested on the basis of experimental work.

Special attention is given to the analysis of silicates and portland cement by J. L. Gilliland. Flame photometry applications to water and soil analyses with emphasis on quality of irrigation water and land classification work are also outlined.

The use of the Perkin-Elmer and Beckman instruments is compared and a procedure is described for the determination of sodium and potassium oxides in portland cement using the Beckman flame photometer. Also various precautions and techniques to avoid error are described.

Scott, Marcy, and Hronas, show that the flame photometer gives results of sufficient precision and accuracy for routine determinations of sodium and potassium in industrial water without the numerous separations required in classical chemical analysis. This method is compared with the gravimetric in tables of data, and the tremendous saving of time is illustrated.

McCoy and Christiansen describe the determination of lithium in portland cement using the flame photometer. A photometer for the analysis of the sodium family in biological fluids or medicines is covered by Wallace and Berry.

Symposium on Structural Sandwiches

The fabrication of lightweight sandwiches for aircraft applications will be featured in a paper by Heebink. Applications in housing, furniture, and transportation will also be considered. Glass cloth, metals, plywood, and paper (for facings) and wood, metal honeycomb, and resin foams (for cores) will be described as well as bonding processes for metal-to-metal adhesives and low-pressure resins. Nondestructive tests and inspection methods may be touched on briefly.

"Paper Honeycomb as a Core for Structural Sandwich Construction," by Kuenzi discusses types of papers and test methods used to determine the mechanical properties of high-strength paper honeycomb core materials for aircraft sandwich

constructions. Descriptions of various paper core materials for houses are given. Mechanical and physical properties such as thermal insulating and decay resistance are described.

A paper by Pajak, "Aluminum Honeycomb Sandwich Construction," defines terms, points out core requirements, describes aluminum honeycomb core, explains various design considerations, includes fabrication procedures, and discusses aircraft floors, doors, partitions, wing panels, fuselage sections, and equipment panel applications.

A discussion of the strengths of sandwich construction in terms of component mechanical properties will be submitted by Norris. He will describe three types of recognized failure: (1) failure of materials due to direct loads imposed, (2) failure of the core or the bond between the core and the facings due to elastic facing instability, and (3) failure of the core due to transverse shear. Design criteria will be suggested.

"Sandwich Construction in the Elastic Range," by March, summarizes theoretical results of the elastic behavior of structural elements made of sandwich material. From these results on orthotropic materials, those for isotropic can readily be obtained. The authors plan to include extensive references to sandwich construction literature.

A review of the last ten years development will be made by Rapp in his paper, "Developments in Structural Sandwich Building Panels Using Inorganic Cores." Features which distinguish products for aeronautical and radar uses from those for building applications are emphasized. One approach to design of fire-resistant compositions having a thickness, strength, and stiffness, and cost compatible with building requirements is described. Data on experimental development, testing, and trial applications of potentially commercial products are included.

Symposium on Acoustical Materials

The history of the acoustical materials industry over the past 30 years, its present size, and the types and uses of its products will be covered in a paper by Waterfall. The importance of standardization will be emphasized. Mr. Waterfall has also written a paper, "Combustibility of Acoustical Materials," which points out the existence of a wide variety of products having all degrees of combustibility. There is little evidence, according to the author, that any of the numerous suggested methods for measuring combustibility show actual life hazard in event of fire. Data will be presented and a plea will be made that present standardization work be continued until a simple test method is developed and a realistic method of classifying materials adopted.

A description of the variables upon which material surface sound absorption coefficients depend is made in the paper by Sabine, "The Measurement of Sound Absorption." He discusses present methods and their limitations. The author also describes a program of ASTM's Com-

mittee C-20 which has been delegated to Subcommittee I on Sound Absorption and which was instituted to study present and other methods. The program will embrace round-robin testing on sound absorption measurement and utilization of all pertinent research work.

The paper, "Application of Acoustical Materials," by Yerges, will list the problems and existing practices, will define some obvious test requirements, and will point out present-day test methods and application techniques.

Chrzanowski will discuss various aspects of the maintenance problem on acoustical materials. Data will show the effects of amount of paint applied and of different painting techniques on sound absorption. "Breathing," precipitation processes, and direct impingement which are responsible for susceptibility to staining of acoustical materials will be discussed.

Consolidation Testing of Soils

In a settlement study of two earth-filled concrete barricades, consolidation tests made with peat are discussed in a paper by Thompson and Palmer. The test results with load increments acting for a maximum of 36 days are presented; also an analysis of the test data and their application to the estimate of the magnitude and rate of future settlement. Factors related to testing equipment and methods that will influence the results obtained in the consolidation test are included in "Aids in the Interpretation of the Consolidation Test" by Matlock and Dawson.

"Consolidation Testing Applied to Highway Foundations" by Barber presents a description of a test procedure, some requirements of exploration, and methods of calculation. Time-settlement observations of several bridges and fills are compared.

Consideration is given in "The Application of Controlled Test Methods in Consolidation Testing" (D. M. Burmister) to concepts and basic principles. Test conditions and techniques must be made to fit the particular behavior characteristics of the soils and the specific conditions that control or govern behavior of soils under stress. Applications of the methods are illustrated.

Loess is an air-borne earth material which covers vast areas of the central part of most of the major continents of the world. In the United States, it covers much of Nebraska, Kansas, and part of South Dakota. Holtz and Gibbs describe laboratory tests to determine the consolidation characteristics of loess soils, index tests for estimating these characteristics, and the effect of consolidation on other soil properties. When these soils are to be used as foundation for hydraulic structures, special care must be given in foundation design.

Symposium on Surface and Subsurface Reconnaissance:

Details of this symposium and additional features of the 1951 Annual Meeting will be covered in the February BULLETIN.

December Approvals of the Standards Committee

Testing Magnetic Materials; Water Vapor Gaseous Fuels; Load Settlement of Piles

ACTING on the recommendation of technical committees concerned with magnetic properties (A-6), gaseous fuels (D-3), and soils (D-18), the Society, through its Standards Committee, has recently approved some important new standards and tentative revisions. These include revised tests for magnetic materials, an important new procedure for determining water vapor content of gaseous fuels, and a new tentative test to determine the load-settlement relationship for individual piles. The Committee A-6 tentative revisions will appear in the back part of the 1950 Supplement to Part 1 of the Book of Standards. The two new tentatives will appear in the respective Supplements to Parts 5 and 3. Copies of the tentatives will also be available in separate pamphlet form and can be obtained on purchase.

Proposed Changes in Magnetic Testing:

A new section, which specifies that the density of cobalt-iron alloys shall be assumed to be 8.1 g. per cu. cm. in the 35 per cent cobalt-iron alloy and that it will be 8.2 g. per cu. cm. in the 50 per cent cobalt-iron alloy is recommended to be added to the Methods of Testing Magnetic Materials A 34 - 49. In Methods of Test for Permeability of Feebly Magnetic Materials, A 342 - 49, Section I is eventually to be modified to change the permeability limit to "not exceeding 4.00." Section II-A is to be modified to make the "permeability between 1.0 and 4.0."

Section IV on Test Specimens will read as follows (*when the proposals are adopted*):

"The test specimen shall consist of straight bars, rods, wires, all straps of uniform cross-section. The cross-sectional area shall be not less than 0.2 sq. cm. The length shall be not less than 10 cm. and the ratio of length to diameter or equivalent diameter (dimensional ratio) shall be as follows: 'The dimensional ratio at least 20 when permeability is between 1.1 and 2.1; dimensional ratio at least 30 when permeability is between 2.0 and 4.0.' (Equivalent diameter is the diameter of the circle having the same area as the cross-sectional area of the specimen.)

In the Methods of Test for Electrical and Mechanical Properties of Magnetic Materials, A 344 - 49, a new method (Method No. 2 on Interlamination Resistance) is to be added when adopted. This will provide a means of testing single strips of flat-rolled electrical

steel for interlamination resistance under predetermined conditions of voltage pressure and temperature. The method will be particularly suitable for quality control for the application of insulating coatings.

Load Settlement of Piles

Agreement having been reached in Committee D-18 on Soils for Engineering Purposes for a proposed test to determine the load-settlement relationship for individual piles, the Society through the Standards Committee approved the new method with the designation D 1143 - 50 T. The scope indicates:

This method covers a procedure for testing individual foundation piles to determine the relationship between the load applied to the pile and the settlement of the pile.

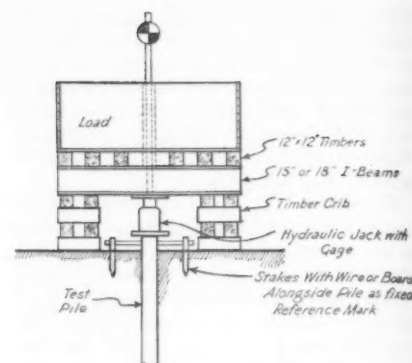
NOTE.—This specification describes only a method of testing a single pile. It does not cover the application of the test results to the carrying capacity of a group of piles or to foundation design in general.

Several methods and equipment are described for applying known vertical loads to the top of the pile and equipment for measuring the settlement.

This new tentative is one of the suggested methods of test which were covered in the comprehensive compilation of Procedures for Testing Soils which book was issued in July. The accompanying illustration shows one of the methods which can be used to apply the load.

Water Vapor Content of Gaseous Fuels

Because of the need for a recognized standard test for water vapor content of gaseous fuels, Committee D-3 on



Gaseous Fuels had as one of its early objectives the drafting of a proposed method. Much time and effort have gone into research leading up to the new tentative method of test D 1142. The scope of the method reads as follows:

Scope:

(a) This method of test employs the measurement of the dew-point temperature for the determination of the water vapor content of gaseous fuels. In general, any properly constructed type of dew-point instrument may be used. . . .

(b) Some gaseous fuels may contain vapors of hydrocarbons or other components which condense into liquid easily. Condensation of vapors other than water may interfere with the measurement of the dew-point temperature. The method is limited to gaseous fuels whose dew-point temperature is not lower than the temperature at which other component vapors condense.

NOTE.—The presence of diethylene glycol vapor, which may result from passage of the gaseous fuel through certain types of dehydration plants, presents a difficulty due to possible formation of glycol fog. No general procedure for eliminating this interference can be recommended at this time.

(c) Theoretically, the method covers a range of water vapor contents from 0-100 per cent; practically, it is limited by the test equipment. In these specifications it is arbitrarily limited (1) to conditions where the dew-point temperature at the test pressure is at least 3 F. lower than the

Approvals by the ASTM Administrative Committee on Standards, December, 1950

New Tentatives

Methods of Test for:

Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature (D 1142 - 50 T)
Determining the Load-Settlement Relationship for Individual Piles (D 1143 - 50 T)

Tentative Revisions of Standards

Methods of Testing:

Magnetic Materials (A 34 - 49)
Permeability of Feebly Magnetic Materials (A 342 - 49)
Electrical and Mechanical Properties of Magnetic Materials (A 344 - 49)

ambient temperature of the testing equipment and the temperature of the pipeline leading the gas from the source to the testing equipment; (2) to conditions where the determined dew-point temperature is not lower than 0 F.; and (3) to conditions at which ice crystals do not form.

It is apparent that the intensive

research work of Committee D-3 extending over several years is now bearing fruit, because a number of new tentative tests have recently been issued. A list of the standards (methods of test) for which Committee D-3 is responsible follows: Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter (D 900 - 48); Analysis of Natural

Gases by the Volumetric Chemical Method (D 1136 - 50 T); Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer (D 1137 - 50 T); Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature (D 1142 - 50 T).

Sigma Phase, Quality Control of Materials, Rapid Identification of Metals—Important New ASTM Books

Standards Supplements Being Printed

VERY soon on our stock shelves will be several new and significant symposiums and compilations. Of these, the Symposium on Sigma Phase, the Symposium on Rapid Methods of Identification of Metals, the Manual on Quality Control, and the Compilation of ASTM Standards on Copper and Copper Alloys are described below.

The 1950 Supplements to the Book of Standards are being printed and it is

expected that those for Part 2 (Non-Ferrous) and Part 6 (Electrical Insulations, Plastics, Rubber) will be available shortly. Those supplements for Part 1 (Ferrous Metals), Part 4 (Paint, Wood, Adhesives, Shipping Containers, Paper), and Part 5 (Fuels, Petroleum, Aromatic Hydrocarbons, Soap, Water, Textiles) should be ready sometime in January-February with Part 3 (Cementitious, Soils, Road and Water-

proofing Materials) becoming available late in February. While these dates are approximate, every effort is being made to get the supplements completed on schedule.

Shipments of the particular supplements to members who have secured the several parts of the Book of Standards will be made as soon as the books are ready. Each member will receive their 1950 Supplements for the Book of Standards parts which he has requested. An order blank has been sent out to all others who have purchased copies of the 1949 Book.

Rapid Identification of Metals

A NEW symposium is now available which provides a rather complete coverage of more recent developments in rapid methods for identifying metals. The papers and discussions in the Symposium on Rapid Methods for the Identification of Metals were presented at the Annual Meeting in 1949, the symposium being sponsored by Committee E-3 on Chemical Analysis of Metals.

The first three papers deal with general principles of spot testing with chemical reagents, electro spot testing and electrographic analysis, and of modern instrumentation for rapid identification of metals. In the remaining papers, specific applications of these methods or of combinations or modifications of them are described.

A breakdown of the symposium is as follows:

- Development, Present State, and Outlook of Spot Test Analysis—Fritz Feigl, Laboratorio da Producao Mineral, Ministerio da Agricultura Rio de Janeiro, Brazil
- Electro Spot Testing and Electrography—H. W. Hermance and H. V. Wadlow, Bell Telephone Labs.
- Instruments for Rapid Metal Identification—R. R. Webster, Jones & Laughlin Steel Corp.
- Separating Alloys by Relative Spot Tests—H. Kirtchik, General Electric Co.
- Rapid Methods for the Identification of Copper-Base Alloys—R. P. Nevers, The American Brass Co.
- Rapid Identification of Metal Finishes—

A. Lewis and D. R. Evans, Western Electric Co., Inc.

Examination of Plated and Protective Coatings by Electrographic Analysis—N. Galitzine and S. E. Q. Ashley, General Electric Co.

A Field Test Kit and Procedure for Use in the Rapid Identification of Some Nickel Alloys and Stainless Steels—Henry B. Lea, Eastman Kodak Co.

Rapid Tests for Identifying Alloy Steels—Elbert C. Kirkham, University of Utah

The above collection of methods and techniques, many of which have not been described previously, should prove of great value wherever rapid identification of metals is required and especially in cases where the testing must be done in the field—where the laboratory must be taken to the sample.

The Symposium (STP No. 98) aggregates 84 pages, is bound in heavy Manila paper, and is priced at \$1.75. The price for members is \$1.35.

Manual on Quality Control of Materials

New Book Replaces Widely Distributed Report on Presentation of Data

A NEW ASTM Manual on Quality Control of Materials has been prepared and is now being published. The Manual is sponsored by Committee E-11 on Quality Control of Materials, and takes the place of the widely used ASTM Manual on Presentation of Data.

The new Manual is organized in three parts. Part 1 essentially covers the presentation of data; Part 2 presents limits of uncertainty of an observed average; Part 3 explains the control chart method of analysis and methods for presentation of data.

Part 1 represents a revision of the main section of the former ASTM Manual which it replaces. This section discusses the application of statistical methods, other problems of condensing the information contained in a single set of n observations, and presenting essential information in a concise form. Special attention is given to types of data such as would be gathered by individuals or committees and presented to the Society, with emphasis on the variability and the nature of frequency distributions of physical properties of materials.

Part 2 represents a revision of Supplement A of the old ASTM Manual which it replaces. In this section is discussed the problem of presenting limits to indicate the uncertainty of the average of a unique sample of n observations. In this part is suggested a form of presentation for use when needed in ASTM reports and publications; the restricted conditions under which this form is theoretically applied are given and the meaning of such limits is explained, and a table is given to permit ready calculation of 99 per cent, 95 per cent, and 90 per cent "confidence limits" for various sample sizes. Working rules are presented regarding the number of places to be

retained in computation and presentation of average, standard deviations, and confidence limits. In this revision the generally accepted term "confidence limit" is introduced, and constants for computing 95 per cent confidence limits are added.

Part 3 presents formulas, tables, and

examples useful in applying the control chart method of analysis and presentation of data as mentioned above. This method requires that the data be obtained from several samples or that the data be capable of subdivision into subgroups on the basis of relevant engineering information. The principles of

this part are discussed largely in terms of the quality of materials and manufactured products.

The ASTM Manual on Quality Control of Materials (*STP No. 15C*) is 100 pages. The heavy Manila-bound Manual is available at \$1.75, the ASTM members' price being \$1.35.

Sigma Phase Book of Interest to All Concerned with Metals and High-Temperature Effects

IN THE past five years there has been an increasing interest on the part of users and producers of metals for use at elevated temperatures in the occurrence, identification, and effects of sigma phase. This phase had been known to exist in chromium-iron-nickel alloys for over 20 years. But the nature of the phase makes a complete understanding of the conditions which promote its occurrence, as well as the development of methods for controlling the effects on properties of metal at high temperatures, essential to further progress in this field.

The Research Panel of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals had been cognizant of the considerable amount of research going on in problems posed by the existence of sigma. Because of this, the panel organized this symposium which was held at two extensive sessions of the 1950 Annual Meeting in Atlantic City. The numerous papers and discussions are now being published.

In general, the symposium presents the progress, trends, and problems in

connection with sigma phase. Admittedly, further data on this constituent of many of the heat-resistant alloys are desirable, and this symposium does give a quite complete picture of our current information. The papers included in the published form of the symposium (see May, 1950, *ASTM BULLETIN*, page 15, for descriptions) and their respective authors, are as follows:

Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Fe-Cr-Ni Steels—E. J. Dulis and G. V. Smith, U. S. Steel Corp.

Sigma Phase in Several Cast Austenitic Steels—V. T. Malcolm and S. Low, Chapman Valve Manufacturing Co.

X-ray Study of the Sigma Phase in Various Alloy Systems—Pol Duwez and Spencer R. Baen, California Institute of Technology

Sigma Phase in Chromium-Molybdenum Alloys with Iron or Nickel—John W. Putman, N. J. Grant, and D. S. Bloom, Massachusetts Institute of Technology

The Tetragonality of the Sigma Phase in the Iron-Chromium System—L.

Menezes, J. K. Roros, and T. A. Read, Columbia University

The Formation of Sigma Phase in 17 per cent Chromium Steel—J. J. Heger, Carnegie-Illinois Steel Corp.

Sigma Phase and Other Effects of Prolonged Heating at Elevated Temperatures on 25 per cent Chromium - 20 per cent Nickel Steel—G. N. Emmanuel, Babcock & Wilcox Tube Co.

The Formation of Sigma and Its Influence on the Behavior of Stabilized 18 per cent Chromium, 8 per cent Nickel Steels in Concentrated Nitric Acid—R. S. Stewart and Stephen F. Urban, National Lead Co.

Some Notes on the Structure and Impact Resistance of Columbium-Bearing 18-8 Steels After Exposure to Elevated Temperatures—W. O. Binder, Union Carbide and Carbon Research Laboratories, Inc.

Observations of the Effect of Sigma on the Mechanical Properties of Columbium Stabilized Weldments in Austenitic Stainless Steels—F. W. Schmitz and M.A. Scheil, A. O. Smith Corp.

The Occurrence of the Sigma Phase and Its Effect on Certain Properties of Cast Fe-Ni-Cr Alloys—J. H. Jackson, Battelle Memorial Institute

The Symposium on Sigma Phase (*STP No. 110*) is a 204-page book, in heavy paper cover, and is available at \$2.50; the members' price is \$1.85.

Copper and Copper Alloys

THE LATEST edition of the widely used compilation of ASTM Standards of Copper and Copper Alloys will be off press about the time this *BULLETIN* reaches the members. It will include the latest approved form (as of January, 1951) of the ASTM specifications and tests relating to cast and wrought copper and copper alloys, electrical conductors, and numerous non-ferrous metals used in copper alloys. While the compilation is sponsored basically by Committee B-5 on Copper and Copper Alloys, Cast and Wrought, in the interest of completeness and to make the publication as useful as possible, several of the specifications of Committee B-1 on Non-Ferrous Metals are included and a number of other selected specifications on non-ferrous metals in the scope of

Committee B-2 on Non-Ferrous Metals and Alloys. All told, there are over 100 standards.

A number of the standards incorporate changes; for example, there is a clarification of the application of certain types of copper water tube; the mercurous nitrate test is clarified in its application for determining internal stresses only; and because of the interest in the requirements of preparing micrographs of metals and alloys (E 2 - 49 T) this has again been included. A brand new addition involves selected portions of Methods of Preparation of Metallographic Specimens (E 3 - 46 T) which applies to the copper and copper alloy field. Groups of materials and products covered in this latest January, 1951, compilation include:

Copper and copper alloys

Copper-covered steel wire, rods, bars,

and stranded conductors for electrical purposes

Non-ferrous metals (zinc, lead, nickel), phosphor, copper, etc.

Copper and copper alloy plate, sheet, and strip

Alloy wire, rods, bars, and shapes

Alloy pipes and tubes

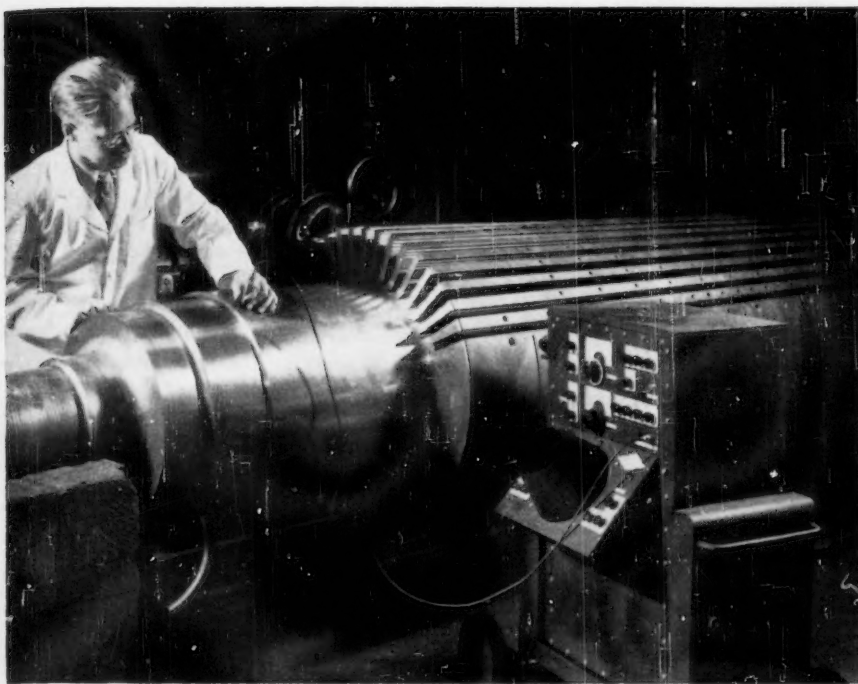
Ingot

Castings

Arc-sealed electrodes

Numerous methods of testing copper and copper alloys

In the past, hundreds of technical people, engineers, and sales representatives concerned with the production and use of materials covered have found this compact compilation a real convenience and will wish to procure the latest edition. It aggregates 524 pages and is bound in heavy paper cover. The list price is \$4.35; to members \$3.25. For those who wish cloth binding this is available at an extra charge of 65 cents per copy.



This shows the supersonic reflectoscope being used at the beginning of a program to test an almost completely machined rotor both longitudinally and diametrically. Mr. Kelman, the author of the paper, is the gentleman shown.

Ultrasonics Symposium Covers Theory, Forgings, Aluminum, Railroad, Non-Ferrous, Adhesive Joints

INCREASING interest in ultrasonic testing and steadily widening applications led ASTM Committee E-7 on Non-Destructive Testing to sponsor a symposium on the subject at the 1949 Annual Meeting with papers by leading authorities. This publication embodies these papers, together with one earlier

paper and two presented subsequently. The papers have just been reviewed prior to publication by their authors, in the light of latest developments; this new publication provides a wealth of up-to-the-minute data and information.

It is indicated in the prefatory state-

ment by H. C. Amsberg that ultrasonic testing is being used extensively today. It is practical and is being applied intelligently by engineers in all branches and in a variety of industries. It is not just another device which can be used for rejecting more material; it can and will aid and abet manufacturing. It is noted that standard equipment is available which will perform very acceptably in the hands of nontechnically trained personnel of ordinary skill.

A list of the papers, with authors, follows:

Introduction—H. C. Amsberg, Westinghouse Electric Corp.

Ultrasonic Wave Propagation in Materials: A Guide to Theoretical Results—Louis Gold, Brown University.

Inspection, Processing, and Manufacturing Control of Metals by Ultrasonic Methods—Carlton H. Hastings and Seymour W. Carter, Watertown Arsenal.

Basic Principles of Practical Ultrasonic Testing—John C. Smack, Sperry Products, Inc.

Ultrasonics in the Heavy Forging Industry—James C. Hartley, Winchester Repeating Arms Co.

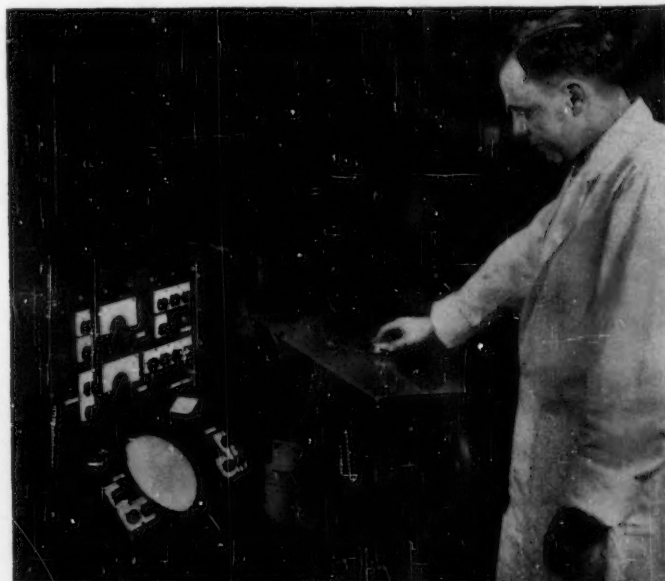
The Application of Ultrasonics to the Fabrication of Aluminum—J. V. Carroll, Aluminum Company of America.

Ultrasonic Testing in Railroad Work—E. D. Hall, Erie Railroad Co.

Ultrasonics in the Electrical Industry—D. N. Kelman, Westinghouse Electric Corp.

Ultrasonic Testing of Bronze Forgings and Ingots—A. Pilch, U. S. Naval Gun Factory.

Pulse Techniques Applied to Dynamic



These two figures show ultrasonics, past and present, first as an art (?), then as a science. In the first the operator is relying on his sense of touch and hearing to detect differences in bond. The product in question is a thrust-bearing runner shoe, and there have been some failures because of faulty bond between the babbit and the steel backing. With latest equipment it is possible to determine very accurately the extent of badly bonded areas.

Testing—J. R. Leslie, The Hydro-Electric Power Co.

The Measurement of Dynamic Modulus in Adhesive Joints at Ultrasonic Frequencies—A. G. H. Dietz, P. J. Closmann, G. M. Kavanagh, and J. N. Rossen, Massachusetts Institute of Technology.

Those who have been working on this

publication, and the committee officers, feel that what has resulted from the close cooperation of members and the authors is a pertinent and valuable publication which will provide latest information on important aspects of this interesting field of ultrasonic testing. This is another in the series of worthwhile books resulting from the work of

ASTM Committee E-7. Another publication on "Non-Destructive Testing in the Economics of Production" is being set in type with publication anticipated in February. Further announcement will be made. Copies of the 120-page Ultrasonics Symposium in heavy paper cover are available at \$2; price to members, \$1.50.

From a Telephone Call

ASTM Radiographic, Cotton Yarn, and Other Charts and Tables

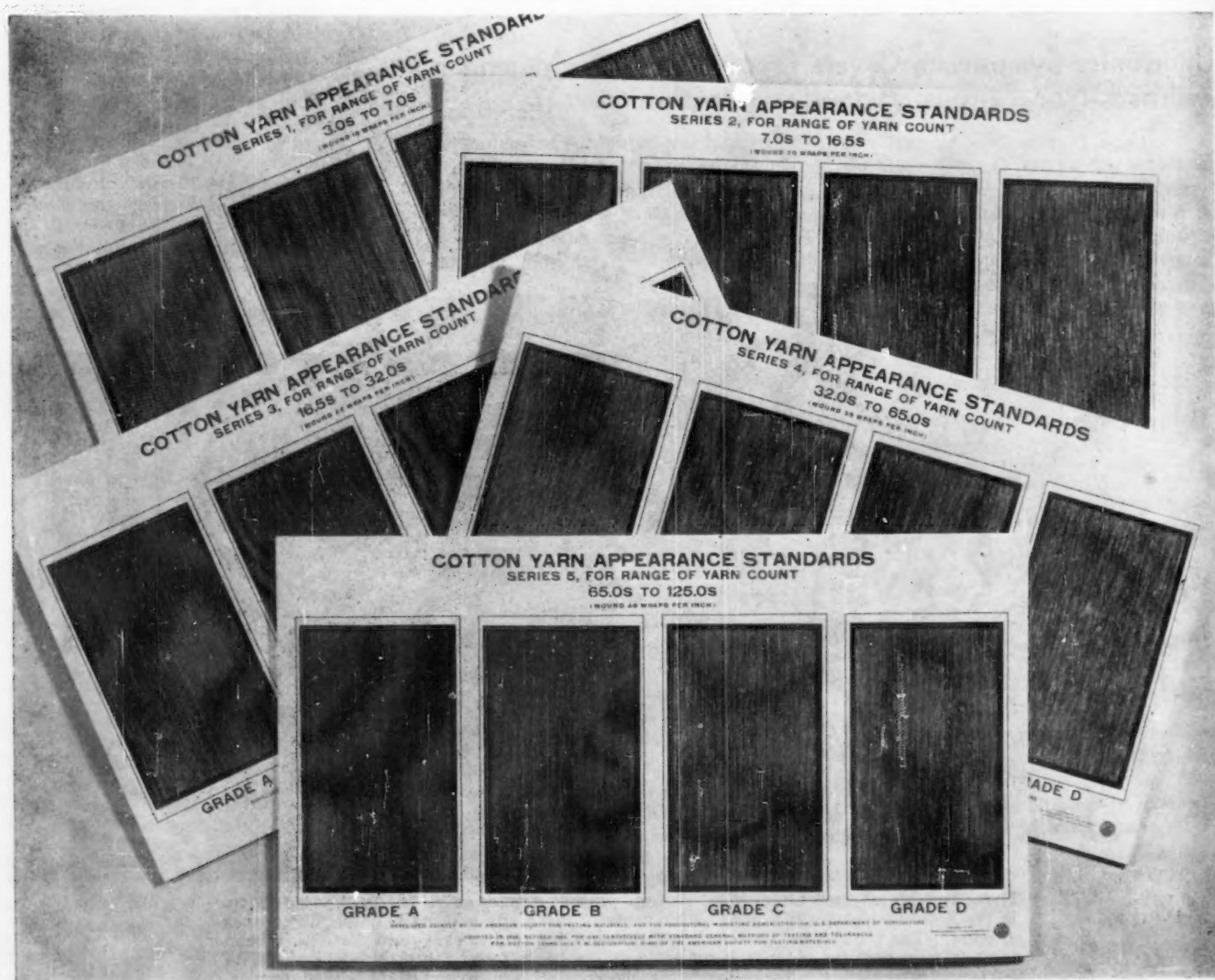
A RECENT emergency telephone call from one of the strategic Government agencies in the Southwest asking for Society help prompts us, now that the help has been given, to comment on a certain group of ASTM publications which to a great majority of our members and readers are probably not too well known. The accompanying photographs illustrate certain of these files of cards and collections of appearance standards, including the ASTM Radiographic Standards for

Steel Castings and the Cotton Yarn Appearance Standards. It is a far cry from the emergency phone call in connection with certain of these standards, to the original considerations which led the Society to embark on publishing the material. No one realized the great service that would be given and the importance which would be ascribed to material of this kind.

Among the publications in this general grouping of tables, charts, and appearance standards are the following:

PAINT PANEL RECORD FORMS
X-RAY DIFFRACTION DATA CARDS
INDUSTRIAL RADIOGRAPHIC STANDARDS FOR STEEL CASTINGS
COTTON YARN APPEARANCE STANDARDS
STEEL HARDENABILITY CHARTS
VISCOSITY-TEMPERATURE CHARTS
TABLES AND CHARTS FOR ENGINE TESTS OF FUELS
VISCOSITY INDEX TABLES
VISCOSITY CONVERSION TABLES
And soon to be added are the VOLUME CORRECTION TABLES

The history of many of these is the same. Some group, possibly a Government agency, developed the original



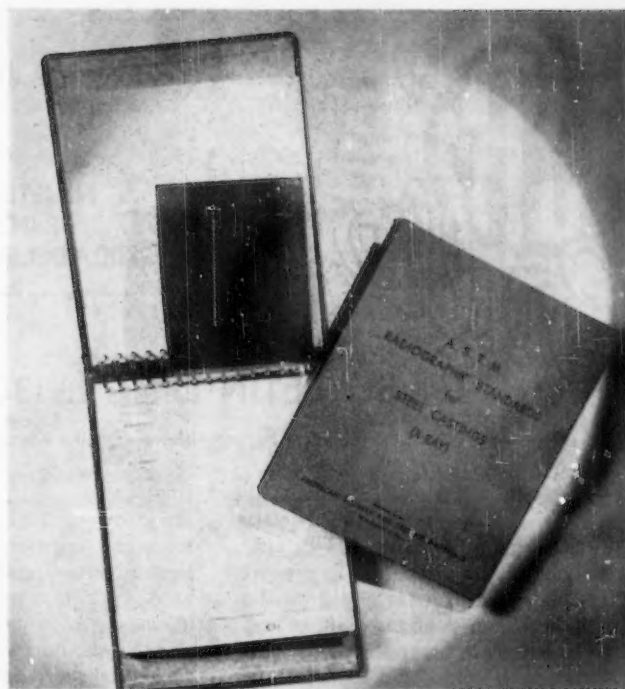
material but was not prepared, or did not see fit, to publish for wider distribution, but was quite ready to have the project taken over by an ASTM committee. The Radiographic Standards for Steel Castings, for example, were originally developed by the United States Navy which had a few sets it could loan to some of the foundries that were producing important material. Our Committee E-7 on Non-Destructive Testing, which included some who had worked on the Navy material, felt that ASTM might do a service in arranging to issue the films, and so the Society took on the task of duplicating very precisely a large number of films, then drafting brief descriptions, the whole to be arranged in a convenient loose-leaf file.

Certainly a limited edition would suffice. But, as with each of the publications in this broad grouping of appearance standards and charts, our guesses have been conservative—and the third edition of Radiographic Standards has just been compiled to fill the orders received. This healthy situation bespeaks the interest of the steel castings industry in quality factors; it has amply justified the work of Committee E-7 in sponsoring the material; and it perhaps may be considered a compliment to the staff men who handled the mechanics of publication and promotion. These reference standards, the X-Ray and the Gamma-Ray being available in separate ring-binders at \$30 per set, or \$55 for both sets, are intended to assist in classifying defects which are occasionally encountered. They are divided into groups according to the type of defect. The reference radiographs can be easily removed from the binders for viewing. Suggestions for classifying castings to be used with the standards are given in the printed Tentative Procedure E 71.

Cotton Yarn Appearance Standards:

The interest in the Cotton Yarn Appearance Standards and their worldwide distribution is also astounding, to say the least. Originally developed by the United States Department of Agriculture, and supplied in limited quantities in connection with determining the quality of cotton appearance, these standards were incorporated in ASTM Methods of Testing and Tolerances (D 180), comprising the five boards illustrated here. Again only a limited number of copies were made available back in 1941, but since that time there has been a continuing demand for the standards, and this in spite of the relatively costly nature of the charts and the fact that several increases in price had to be made. Essentially, the standards consist of twenty 5½ by 10-in.

X-Ray and Gamma Ray Radiographic Standards



photographs representing four grades each of five groups of yarn numbers. It was possible for the Society to issue the boards at first at \$10 per set, but the current price is \$4.50 per board or \$22.50 per set. The photographs have to be very carefully done because of the preciseness required.

Many members and others understand about the Society's *Proceedings*, its *ASTM BULLETIN*, and the world-famous Book of ASTM Standards. The Society has a number of other important publication projects (and problems!), all of which have as their common basic aim providing a service to industry and government.

ASTM Standards for Use in Binders

SOME years ago, on the assumption that a number of the Society members might wish to assemble selected ASTM standards in loose leaf binders—in other words make up their own compilations—the practice was instituted of drilling three holes in all separate standards to fit the standard 6 by 9-in. binder. Special binders were also made available at cost for those interested in assembling separates in this way. Apparently there has been very little interest on the part of members to use the standards in this form, and it is accordingly proposed to discontinue drilling of standards, at least temporarily, since it is an item of some expense and it is somewhat time consuming. If it is found that this proves to be an inconvenience to members, the practice will be reinstituted.

Pacific Area Papers

CONTINUING to keep the membership informed as to the availability of papers which were presented at the First Pacific Area Meeting of the Society in the Fall of 1949, there follows a list of six papers which will be published in the 1950 *Proceedings*.

Session on Fatigue of Metals:

- Fatigue Strength of Steel Through the Range from ½ to 30,000 Cycles of Stress—M. H. Weisman and M. H. Kaplan
- Fatigue Notch Sensitivities of Some Aircraft Materials—H. J. Grover

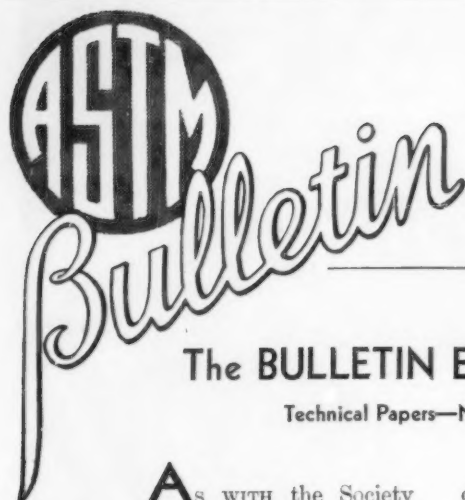
Session on Soils:

- Electrical Resistivity Method Applied to the Investigation of Construction Materials Deposits—E. A. Abdun-Nur and D. Wantland
- Effect of Rock Content and Placement Density on Consolidation and Related Pore Pressure in Embankment Construction—H. J. Gibbs

Session on Cement and Concrete:

- Why Type II Cement?—F. H. Jackson
- Long-Time Study of Cement Performance in Concrete, with Special Reference to Heats of Hydration—G. J. Verbeck and C. W. Foster

In the July and December, 1950, *BULLETINS* data were published describing the availability of other Pacific Area papers.



JANUARY 1951

NO. 171

NINETEEN-SIXTEEN
RACE STREET
PHILADELPHIA 3, PENNA.

The BULLETIN Enters Its 30th Year

Technical Papers—News—For the Members

AS WITH the Society itself, the growth of the ASTM BULLETIN has been a steady one. The present BULLETIN is indeed a far cry from the straight-text, four-page publication first printed under this name in 1921. The changes in both format and content have been striking, and it may be appropriate now at the mid-century to review the more important of these changes.

As the official organ of ASTM, the BULLETIN has developed in form and content throughout the years as the Society's needs have changed and expanded. The first issue, coming 19 years after the Society was incorporated, stated as the reason for publication the necessity for prompt and regular dissemination of news throughout the membership, and the necessity for close cooperation between the members and Headquarters, since ASTM work was a year-round business—and could not possibly all be concentrated at the Annual Meeting. This is still the major function of the BULLETIN. Side-by-side with the statement of purposes in the first issue was a news article concerning the then forthcoming Annual Meeting, which stated among other things that the meeting was to be held in the New Monterey Hotel, Asbury Park, N. J., from June 20 to 24, inclusive. Now, in 1951, news of the Annual Meeting still retains as prominent a place in the BULLETIN and in the minds of Society members.

With the April, 1926, issue, advertising was included. In explanation, the editor stated that the previous five years of publication had seen considerable expansion of Society membership and that a larger BULLETIN containing more news would be very desirable. Advertising income would permit a more complete job of keeping members informed. Advertising was limited, as it is today, to that dealing with laboratory supplies, instruments and apparatus, technical books, and a

directory of commercial testing and research companies. It was also noted that advertisements of this type would most certainly prove interesting and informative to members, and its inclusion should be a further service. This viewpoint still prevails. It is interesting to note that a number of advertisers using the first issue still consistently appear in the BULLETIN.

Ten years later, in 1936, the BULLETIN carried its first technical papers. Then as now, the emphasis in selection of papers was on technical content, general interest, timeliness, and authoritativeness. The diversity of the papers may be readily appreciated from a quick glance at the index of BULLETIN articles printed in the December, 1950, issue. Their high standard of quality is maintained by the Papers Committee which gives these papers the same close editorial scrutiny given the papers published in the Society's

yearly *Proceedings*. For members who wish to make a complete file of technical papers they have been numbered consecutively with a "TP" number in addition to a page number. They are also completely indexed in the *Proceedings*.

It has been the practice to include enough technical papers in each BULLETIN to provide substantial fare, but not so many as to make the publication bulky or difficult to read shortly after it is received.

In the years between 1936 and 1940, the text material more than doubled, and in January of 1940 the BULLETIN contained enough material to justify setting aside a separate page for the table of contents. This issue also unveiled the present familiar cover and contained the editorial explanation that the cover had been chosen because it had been impossible to design a cover symbolically indicating all of the very diverse activities of the Society.

In further recognition of its responsibility to inform its members, the BULLETIN introduced its "Notes on Laboratory Supplies" section in December, 1947. New manufacturer's literature, releases on new instruments, and news of instrument companies are carefully reviewed and passed on to the members through this column.

In the course of gradual growth and more frequent publication, certain issues of the BULLETIN have come to be associated with certain editorial features. The January issue contains a review of the year's accomplishments; the April issue reports on "Committee Week"; the May issue contains the advance program of the Annual Meet-



The Eight Issues of
the ASTM Bulletin for
1950, Totaling 774
pages

ing, and the July issue contains a review of the accomplishments of the Annual Meeting. The September issue contains a complete over-all recapitulation of the research and standardization projects in progress in ASTM Committees; while the October issue features a review of the Actions on Standards. This listing graphically in-

dicates the continuous year-round nature of Society work.

In the past decade alone, the BULLETIN has grown from a publication totaling 320 text pages and 72 advertising pages, to the 1950 total of 603 text pages and 171 advertising pages.

The BULLETIN has been on an eight-issue basis since July, 1949. This is a

somewhat unusual publication schedule, but it fits into our printing program well.

A history of our official organ, even as incomplete a one as this, is in a sense a history of the Society itself. As the Society has grown and broadened its field of interest, the BULLETIN has kept step—striving always to be of maximum service to the Society and its members.

Teamwork and the ASTM*

A Message from President Markwardt

Dear Fellow Member of the ASTM:

Mine today is the privilege of bringing you New Year's greetings for 1951—greetings from your officers and from your loyal headquarters staff, as well as my own sincere greetings and best wishes.

The New Year is traditionally a time for drafting resolutions, for taking inventory, and for self-appraisal. It is doubtful if there has been in present memories a time of greater uncertainty and of greater economic and political concern than faces us at present. It is a time that demands sound leadership and clear thinking. It is a time when these lines from Josiah Gilbert Holland ring even more challengingly than in the era in which they were written:

God, give us men! A time like this demands
Strong minds, great hearts, true faith,
and ready hands;
Men whom the lust of office does not kill;
Men whom the spoils of office cannot buy;
Men who possess opinions and a will;
Men who have honor; men who will not lie;
Men who can stand before a demagogue
And damn his treacherous flatteries
without winking!
Tall men, sun crowned, who live above
the fog
In public duty and in private thinking.

A broad appraisal of the ASTM at this New Year's time reveals a wealth of assets: a smooth running and efficient organization that has in a short half century attained a vital place of service in our national economy; a loyal, industrious and ably managed staff, broadly directed by a capable Board with whom it is a pleasure to work; a fine headquarters building, largely financed by the members; an ever-growing volume of standard specifications and methods of test now numbering over 1700,

that together with other technical information comprises the product of our production line, and as such provides much of our revenue; a sound financial condition, with a balanced budget; but most important of all, a diversified technical membership, as a result of whose industry our standards are produced, and without whom our Society obviously could not exist.

I would be remiss if I did not take this opportunity to extend to each of you, fellow members, my sincere appreciation of your contribution to the work of the Society. The extent of this contribution is all the more impressive when one realizes that at the last annual meeting alone, some 23 sessions and over 500 committee meetings were held.

The "give and take" that is so essential in our committees is what represents the invaluable teamwork of the ASTM. There is still much food for thought, in this age of mechanical power, in this philosophy of rural America called Horse Sense:

A horse can't pull while kicking.
This fact I merely mention.
And he can't kick while pulling,
Which is my chief contention.

It's teamwork on the job that counts,
The working together as one;
It's teamwork as the pressure mounts,
That insures the job will be done.

I would certainly be over-optimistic if I left the impression that the ASTM had no problems or no needs. These are ever present in an active organization. Our greatest need is for increased membership—as an active organization we must continue to grow. I am glad to report that the ASTM membership is still growing, but too small a portion of our budget is from membership dues. Despite what our headquarters staff and officers can do, increase in membership still depends largely on the help of members and of the committees.

Many hands can accomplish much to this end.

I have referred previously to the uncertainties we face today. Yet in spite of these uncertainties, there are some things of which we can be quite sure. Experience has shown that while the ASTM plays an essential part in our peacetime economy, it is even more important in times of emergency. ASTM standards played a vital role in World War II, and this extended use of standards was accompanied by an increase, not a decrease, in Society membership. In the present emergency we are already facing critical material shortages. Again judging by experience, this calls for revisions of standards and methods, and presages increased technical committee work. The Board of Directors, in recognition of this situation, some time ago appointed a special committee to contact Government agencies and to offer the services of the ASTM.

As the Society's work progresses, its activities continue to expand. The development of new materials and new processes, and the encountering of new problems, require that the Society be ever alert to meet its opportunities for service. As in previous years, there took place during the year the organization of a number of new technical committees, and more are under consideration.

In conclusion, it bears repeating that the ASTM has proved itself a highly important and effective agency for technical guidance in its field. With the teamwork of the membership it can, in an even larger measure, meet the challenge for service—service to the Nation and to its industry.

Sincerely yours,

L. J. MARKWARDT, President

Madison, Wisconsin
January 2, 1951

* This New Year's Message from President Markwardt was sent to each member of the Society early in January. The editors thought that many of the BULLETIN readers who did not receive the Message directly would find it of much interest.

70 Student Membership Prize Awards Established

Members Underwrite Student Recognition

As a result of a recent communication to the members, a number of Student Membership Prize Awards have been established at leading universities and technical schools. This project provides that interested members of the Society can underwrite any number of ASTM student memberships at \$2 each, these being awarded to outstanding engineering and technical students, selection being made usually by the faculties of the respective schools.

This award plan has been in effect for many years, but during the recent World War became somewhat of a casualty because of the speed-up in engineering training. There follows a list of the schools at which prize award plans are to be in effect, with the names of the donors, the number of awards totaling 70:

University of California, Berkeley, Calif.—Bertram F. Kline
Iowa State College, Ames, Iowa—Hugh P. Bigler
Massachusetts Institute of Technology, Cambridge, Mass.—John R. Freeman, Jr.
Worcester Polytechnic Institute, Worcester, Mass.—Simon Collier
Stevens Institute of Technology, Hoboken, N. J.—Martin Mahler
Columbia University, New York, N. Y.—James T. Kemp
Rensselaer Polytechnic Institute, Troy, N. Y.—Robert J. Painter
Ohio Northern University, Ada, Ohio—Robert S. Armstrong
University of Toledo, Toledo, Ohio—C. R. Muenger
University of Oklahoma, Norman, Okla.—Two groups, Cecil W. Armstrong and W. A. Schlueter respectively

University of Pennsylvania, Philadelphia, Pa.—C. Laurence Warwick
Ecole Polytechnique, Paris, France—Comptoir Franco-Belge des Tubes
University of Lausanne, Lausanne, Switzerland—Anonymous

This plan has been reinstituted because it is an excellent way of recognizing the work of outstanding students in engineering, particularly in courses which tie in with ASTM work involving materials; it acquaints a deserving and influential group of young potential engineers with the work of the Society through the publications they are furnished; and the plan provides a means whereby an interested ASTM member can at a very modest financial outlay underwrite a cause which is worth while from the standpoint of the student, the school, and the Society.

Other members who are interested are urged to consider this Student Membership Prize Award plan. The number of awards sponsored by various individuals ranges from one to ten.

ASTM Photographs in International Exhibit

QUITE a number of the technical photographs shown in the 1950 ASTM Photographic Exhibit in Atlantic City appeared in the Technical Exhibit which was part of the 1950 International Exhibit of Photographs sponsored by the Photographic Society of America.

The International Exhibit was held in Baltimore, the Technical Division being at The Peale Museum. The following statement appears in the Introduction to the List of Technical Exhibitors.

"This Technical Exhibit includes a group of photographs selected from the 1950 Photographic Exhibit of the American Society for Testing Materials. These pictures show the importance of such techniques as electron micrography, photomicrography, stress analysis, and high-speed photography in control testing and industrial research. The prints were made available for this Exhibit through the courtesy of the ASTM Committee and of the photographers concerned."

There will not be an ASTM Photographic Exhibit (or Apparatus Exhibit) at the 1951 Annual Meeting in Atlantic City, but both a Photographic and an Apparatus Exhibit are planned for 1952 in New York City. There has been much interest in the Photographic Exhibit. Many of the prints are being published in the ASTM BULLETIN from time to time.

Schedule of ASTM Meetings

DATE	GROUP	PLACE
January 18	Sub VIII of Joint Committee on Filler Metal	New York, N. Y.
January 30-31	Committee B-5 on Copper and Copper Alloys	Philadelphia, Pa.
February	Committee D-6 on Paper and Paper Products	New York, N. Y.
February 4-9	Committee D-2 on Petroleum Products and Lubricants	Washington, D. C.
February 5	Cleveland District	Cleveland, Ohio
February 5-7	Committee A-1 on Steel	Cleveland, Ohio
February 20	Philadelphia District	Philadelphia, Pa.
February 20	St. Louis District	St. Louis, Mo.
February 22	Committee D-1 on Paint, Varnish, Lacquer, and Related Products—Sub on Printing Ink	New York, N. Y.
February 26-28	Committee D-1 on Paint, Varnish, Lacquer, and Related Products	Washington, D. C.
February 27	Committee E-12 on Appearance	Washington, D. C.
March 2	Nominating Committee	Philadelphia, Pa.
March 5-9	SPRING MEETING AND COMMITTEE WEEK	Cincinnati, Ohio
March 14-16	Committee D-13 on Textile Materials	New York, N. Y.
March 19-20	Committee D-12 on Soaps and Other Detergents	New York, N. Y.
March 22	Philadelphia District	Philadelphia, Pa.
March 27-28	Committee D-20 on Plastics	Washington, D. C.
March 28-30	Committee D-9 on Electrical Insulating Materials	Washington, D. C.
March 29-30	Committee D-15 on Engine Antifreezes	Washington, D. C.
April 16-17	Committee D-10 on Shipping Containers	Atlantic City, N. J.
April 19-20	Committee B-1 on Wires for Electrical Conductors	Washington, D. C.
April 21	Committee C-21 on Ceramic Whiteware	Chicago, Ill.
April 23-24	Committee D-14 on Adhesives	Washington, D. C.
May 2-3	Committee D-21 on Wax Polishes and Related Materials	Chicago, Ill. and Racine, Wis.
May 21-22	Committee E-11 on Quality Control of Materials	Cleveland, Ohio
June 18-22	ANNUAL MEETING	Atlantic City, N. J.

ASTM DISTRICT ACTIVITIES

President Markwardt Speaks at New England, Chicago and Western New York-Ontario District Meetings

Covers Program in Forest Products Research

AT THREE interesting meetings under the auspices of ASTM Districts in New England, Chicago, and Western New York-Ontario, the meetings being held in Cambridge, Chicago, and Buffalo, respectively, President L. J. Markwardt gave a most interesting address on "Highlights of Progress in Forest Products Research." He made effective use of samples of many of the newer products that have resulted from the intensive research activities under way, as, for example, numerous samples of the newer sandwich constructions, some with honeycomb cores of paper, with various types of facings such as aluminum—all of these constructions embodying light weight with great strength. He also showed samples of dense, beautiful woods made by impregnation of resins and subjection to heat and pressure.

Mr. Markwardt also referred to the economies to be secured through laminated constructions and exhibited as an example a section of a built-up oak boat keel that had undergone many cycles of

alternate wetting and drying without showing deterioration of its bonding material. He also referred to the many improvements in processes that had been effected through research, one of the latest being a new circular saw that would greatly reduce the waste of timber by having a much narrower saw kerf. As a novelty, he exhibited samples of molasses and sugar that had been made from wood, the processes of manufacture being still rather expensive but so much improved that these products have been made available if necessary.

It is expected that his paper will be published in a forthcoming issue of the ASTM BULLETIN. President Markwardt's talk at the Boston meeting was preceded by a dinner at the conclusion of which Chairman Lester introduced representatives of a number of other organizations who had been invited to the meeting and called upon the Assistant Executive Secretary, who was present representing the Home Office.

For the New England meeting, those who had an active part in planning

and carrying through were District Chairman H. H. Lester, Watertown Arsenal, Secretary C. G. Lutts, Boston Naval Shipyard, and Program Chairman E. N. Downing, General Electric Co.

In Buffalo three District Officers, Messrs. L. F. Hoyt, Chairman, Joseph Gentile, Vice-Chairman, and Fred A. Webber, Secretary, handled the meeting arrangements at the Hotel Sheraton with Past-Chairman Thomas L. Mayer closely cooperating.

In the Chicago program, W. L. Bowler made arrangements, closely cooperating with the Western Society of Engineers (WSE) with which this meeting was jointly sponsored using the very fine WSE meeting facilities. WSE Vice-President Becker opened the meeting with Mr. Bowler acting as Technical Chairman. Other District Officers, including Chairman J. E. Ott, Secretary D. D. Rubek, and Vice-Chairman George Stryker cooperated closely.

In Chicago and New England there were about 100 present at each technical session; in Buffalo about 40 (the latter undoubtedly being affected by all-afternoon snow flurries).

Are We Conserving Our Forests and Streams

L. A. Danse Stresses Urgency of Conservation—Vitality Important to Industry

IN A very forceful address, L. A. Danse, Supervisor Materials & Processes, Production Engineering, General Motors Corp., stressed the vital necessity of conserving our forests and streams, emphasizing particularly industry's stake in good sources of process water. Concerned with the subject not only from his industrial work but also personally, Mr. Danse was applauded vigorously after his address at the ASTM Philadelphia District meeting in the Franklin Institute on November 30. He spoke without notes for 55 minutes.

Preceding the technical session, Herbert W. Stuart, Director of Quality Control, United States Pipe and Foundry Co., gave an interesting coffee talk to a group of about 50 present at the informal dinner. In essence, he gave a thumbnail sketch of the cast iron industry indicating the very large tonnage of products, the great extent of the work, and then mentioned some of the newer developments. He praised the work of ASTM in making available suitable specifications and tests to insure a high quality of product. District Chairman

A. O. Schaefer presided at the dinner and opened the technical session. He then asked technical chairman Francis G. Tatnall, Manager of Testing Research, The Baldwin Locomotive Works, himself vitally concerned with conservation particularly from the standpoint of forestry, to introduce Mr. Danse.

Born 100 miles west of El Paso, Mr. Danse early realized the vital necessity of water supplies. From his subsequent industrial experience, and particularly now, since he is concerned with materials and processes, he has a keen perception of the situation. The speaker cited the falling water tables in midwest industrial states and then praised such projects as the Muskingum control work and others in Ohio.

He seriously questioned the value of dams and impounding reservoirs when associated work, to provide reforestation or to conserve existing ground protection, is ignored. The usual result is reservoir silting.

Many branches of industry have as their No. 1 problem the securing of suitable process water in sufficient

amount. In electroplating, for example, impurities in the wash waters are extremely harmful.

Some of the interesting uses of water were cited, for example, as barriers and an implement to remove excess paint in paint spray booths.

Today new plant location is governed very largely by source of supply of materials, including water, while other factors, such as the labor supply, are of secondary consideration. Management has frequently to supplement the water supply for communities where a plant demands so much water that existing wells in the neighborhood are made inoperative.

Mr. Danse was as effective a speaker as has appeared at any of the Philadelphia meetings, and the group of about 75 present to hear him were well repaid for their attendance. In commenting on the meeting, someone said, "Mr. Danse did an excellent job, his words really aroused active interest. It is certainly gratifying to know that people of his prominence are promoting conservation efforts and are trying to bring about a realization of the importance of our natural resources."

Gillies Speaks in Cleveland on Iron Ore

Private Industry Has Brought About Reassuring Picture of Ore Supply

IN A most interesting and informative address in Tomlinson Hall, Case Institute of Technology, Cleveland, Ohio, on November 14, Donald B. Gillies, Chief Consultant on Mining, Republic Steel Corp., Past-President AIME, and President, Lake Superior Iron Ore Assn., gave impressive evidence of the intensive work carried out by private industry in assuring this nation of adequate iron ore supplies. In his own words, "the discoveries which I have described remove for an indefinite period of many, many years any hazard of ore shortage which might handicap the development of this nation. We can look forward to the future with assurance and a feeling of confidence."

This meeting, attended by about 100 members and friends of ASTM, was sponsored by the Cleveland District which, for many months, had been planning to have Mr. Gillies speak to them on this vital subject. Prior to the technical session, an informal dinner was held with upwards of 80 present. Ray T. Bayless, Chairman of the District, presided, and Earl C. Smith, Republic Steel Corp., served as technical chairman and with Mr. Gillies participated in answering questions. R. B. Textor, the Textor Laboratories, arranged the meeting as Chairman of the District Program Committee. Through the courtesy of an associate of Mr. Bayless, Mr. Walter Morrison, Director of Public Relations, American Society for Metals, there follows an interesting statement highlighting Mr. Gillies' address.

The Hot Stove League in the iron and steel business has been keeping us fairly warm during the rapidly thawing cold war. This insulation against a lot of speculative fears has intrigued many of us, but it is to be doubted that the conclusions are based on the right kind of information.

Donald B. Gillies, a veteran in about all the historical and geographical periods of iron and steel development, speaks for Republic Steel as his producer's chief adviser on mining. He assayed the ore situation for the United States—and thus the real kernel of America's future production of things made of metal—in his address.

There was little, if any, speculation in the Gillies talk. Potentials, yes. But potentials on our ability to tie together the three-link chain that spells production of pig iron—namely (1) the existence and availability of ore, (2) the moving of the ore to the blast furnace, and (3) the construction and manning of the blast furnace itself.

Mr. Gillies did not tell his listeners how to alloy metals, or to what use they should be put. But he made it clear that alloying, designing, treating, and fabricating are all steps which must follow pig iron production. To get pig iron, you must have ore; you must get it to the producer's docks; you must have the blast furnace built and in operation.

The first of the three-link chain of pig iron output, according to the evidence presented by Gillies, is much farther along than most of us think. Republic, together with U. S. Steel, Bethlehem, Jones & Laughlin, Armco, Inland, and others can account for enough available ore to boost pig production to fantastic figures. Scores of millions of dollars have already been taken out of private funds to insure the presence and the availability of all the ore we need for national security. More millions of private money is earmarked for the remaining two links in the production chain.

Mr. Gillies reminded us that steel producers cannot pull blast furnaces out of a hat. Moreover, he called attention to the undeniable fact that both of the two links remaining to be joined after ore is found and made available for our use require steel that may well be in the ore rather than in pigs and billets. In other words, transporting the ore from the earth deposits in Labrador, Africa, South America, or any other point takes rails, gondolas, ships, and power plants. All of these are made of steel.

The problem, then, is not, as some seem to guess, just a matter of *plant expansion*. To increase our steel producing capacity of 99.4 million net tons in 1950 to 100 million net tons in 1951 means an increase in all the three links that spell output of pig iron.

Public concern over a shortage of ore is not justified, Gillies told the ASTM listeners. Doubts, if any, should be centered on the problem of producing enough pig iron (from ore readily transferrable from the mine to the producers' dock) to build the railroads, the ships, and the motive power needed for the hauling.

In Chile, Gillies pointed out, Bethlehem has two deposits which total 105,000,000 tons of reserves, ranging in quality from 50 to 60 per cent iron content. Republic has nearly 123,000,000 tons of ore reserves in Liberia, with quality up to 66 per cent iron content.

In Venezuela, U. S. Steel has more than 1,000,000,000 tons of ore reserves of high-grade ore. Bethlehem has up to 60,000,000 tons in that South American country, reported to be 60 per cent iron.

Six American companies and several Canadian companies share in holding close to 400,000,000 tons of 60 per cent iron ore in the Quebec-Labrador region.

In addition to all this, Gillies reported that there will be a 25,000,000 gross ton increase in our domestic ore supply with the completion of the taconite operations in the Great Lakes region.

It all adds up to optimism. Mr. Gillies is not given to painting with deceiving colors, but his documented address made an important group of iron and steel men feel a little more confident about the future. It also pointed out where the trouble lies.

WALTER MORRISON

C. H. Fellows, Detroit Edison Co., currently a member of the ASTM Board of Directors, represented the Society Officers at the meeting; and, also present for a portion of the time was Frank G. Steinebach, Penton Publishing Co., Chairman of the ASTM Administrative Committee on District Activities.

District Meetings Ahead

Composites and Structural Sandwich Constructions, Latest News on Titanium, Techniques in Development of Engineering Materials to be Features

AS THIS BULLETIN nears press, information has been received concerning further district meetings to be held. One is planned in Philadelphia on January 16 when President Markwardt will present the technical address on "Composites and Structural Sandwich Construction," while in New York, on January 12, two leading authorities on titanium will report on latest developments in the use of this phenomenal metal, namely, T. W. Lippert, Titanium Metals Corp. of America, and N. E. Promisel, Bureau of Aeronautics, U. S. Navy. Then, on February 20 in Philadelphia, J. D. Nisbet, General Electric Co., will be the principal speaker on the subject "New Techniques in Developing Engineering Materials, Particularly Metallic Materials."

At the Philadelphia meeting in January, Executive Secretary C. L. Warwick will speak at the main session on "Highlights of Current ASTM Work in Materials" and the Coffee Speaker is E. J. Albert, President, Thwing-Albert Instrument Co., and President, *Pro Tem*, Scientific Apparatus Makers' Assn. Mr. Albert will discuss Philadelphia as an historical and currently vital location in the production of scientific instruments and laboratory supplies.

President Markwardt to Speak in Cleveland

ON MONDAY, February 5, during the meetings of Committee A-1 on Steel which are to be held at the Hotel Cleveland, Monday through Wednesday inclusive, President L. J. Markwardt will speak at a meeting sponsored by the Cleveland District on the subject "Development and Trends in Lightweight Composite Con-

struction." At the dinner preceding the technical session, Norman L. Mochel, Manager, Metallurgical Engineering, Westinghouse Electric Corp., Philadelphia, Pa.; Chairman of the Steel Committee, will give the coffee

talk discussing some aspects of the current critical metals situation.

The Cleveland District thought it would be quite appropriate, while the Steel Committee is in town, to arrange a

district meeting and a President's Night. All those in the Cleveland District will receive a direct mail notice of the meeting, and all members and committee members are cordially invited to attend.

ASTM TECHNICAL COMMITTEE NOTES

Inter-Society Color Council to Meet in February

ASTM Committee on Appearance Will Sponsor Program on February 27

THE 1951 annual meeting of the Inter-Society Color Council, of which ASTM is one of twenty Member Bodies, will be held on February 28, 1951, in Washington, D. C., with headquarters at the Wardman Park Hotel.

The National Bureau of Standards, in celebration of its fiftieth anniversary, has invited a number of scientific societies to meet there during the week of February 26. ASTM Committee D-1 (Paints) meets February 26-28, Committee E-12 (Appearance) meets February 27, the Inter-Society Color Council meets February 28, and the Optical Society of America from March 1 to 3, inclusive.

The ISCC meetings will consist of three sessions. In the morning there is a Discussion and Business Session; the afternoon session will consist of a symposium of reports and demonstrations on Color in Government, a program arranged under the chairmanship of Dr. Deane B. Judd. In the evening the Photometry and Colorimetry Section of the National Bureau of Standards will hold Open House for the group.

The afternoon session, Color in Government, will consist of the following three parts, and as far as possible speakers will use demonstrations to

show what the problems are and how they are being answered:

- I. Color Standards for Paint
- II. Color Problems in the Armed Services
- III. Miscellaneous Color Studies in Government

All persons interested are invited to attend.

ASTM Committee E-12 to Meet

ASTM, one of the Member Bodies of the Council, has, of course, a great many committees concerned with appearance problems, most of which deal with specific products. For example, D-1 on Paints; D-2 on Petroleum Products and Lubricants; with others for textiles, plastics, etc.

Committee E-12 was organized to cut across all groups within ASTM that deal with appearance problems. This committee will hold its 1951 meeting at the National Bureau of Standards on February 27, the day before the ISCC meeting.

Appearance, Its Description, Measurement, and Specification, a Study of What is Being Done by Others and What Needs to Be Done

The theme of the E-12 meeting will be "Appearance, Its Description, Measurement, and Specification, a Study of What Is Being Done by Others and What Needs to be Done." The morning session will open with a report regarding ASTM requirements for appearance terminology and will be followed by reports of work now completed or well under way by other groups that should be taken into consideration in planning the work of E-12. These reports, as far as possible, will be presented by someone closely connected with the work of the sponsoring group: ISCC, Color Terms Report; OSA, the Colorimetry Committee report; ICI, terminology and standards relating to appearance; ASA, Standards relating to the appearance of materials; IES, Photometric Nomenclature and Standards; AATCC, methods and standards for appearance properties of textiles. In the afternoon the work of ASTM committees on appearance properties of materials will be discussed.

A brief business meeting will be followed by one of Ralph Evans' very fine lectures, "Some Psychological Factors of Appearance." This lecture, given as a part of an IES Color Committee Report at French Lick in September, 1949, is so suitable to the E-12 discussion that a repeat performance has been requested.

Favorable Support of Exposure Test Site Program

DETAILS (including the increased scope of work and necessary expenses relative to test site facilities and maintenance) of the proposed ASTM Exposure Test Site Program were published in the October ASTM BULLETIN.

Subsequently, reprints of this article with letters of invitation to participate in this program were mailed to 500 companies representing a wide variety of industries interested in atmospheric corrosion. The initial response to this general request for financial support has been very gratifying. As of January 1, 1951, 150 replies had been received; more than one third of these replies were

favorable or indicated that active participation is under consideration. Over \$12,000 has been received since the middle of November, and the total contributions at the end of 1950 amounted to \$62,650.

The 48 companies now supporting this test site program are as follows:

Acme Steel Co.
Allegheny Ludlum Steel Corp.
Allied Research Products, Inc.
Allis-Chalmers Manufacturing Co.
Aluminum Company of America
American Bureau of Shipping
American Chemical Paint
Apex Smelting Co.
Armco Steel Corp.

Babcock & Wilcox Tube Co.
Baltimore & Ohio Railroad Co.
Bell Telephone Laboratories, Inc.
Bethlehem Steel Co., Inc.
Briggs & Stratton Corp.
Carpenter Steel Co.
Catalin Corporation of America
Christiansen Corp.
Continental Steel Corp.
Crucible Steel Company of America
Dow Chemical Co.
E. I. du Pont de Nemours and Co., Inc.
Eastern Stainless Steel Corp.
W. P. Fuller and Company
General Electric Co.
Grand Rapids Varnish Corp.
Granite City Steel Co.
Gulf Research and Development Co.
Hercules Powder Co.
Hunter Douglas Corp.
International Nickel Co., Inc.

Irvington Varnish and Insulator Co.
 Joslyn Manufacturing and Supply Co.
 Jones and Laughlin Steel Corp.
 Lockheed Aircraft Corp.
 Lukens Steel Co.
 National Cash Register Co.
 New Products Corp.
 Nuodex Products Co., Inc.
 Pure Oil Co.
 Republic Steel Corp.
 Revere Copper & Brass, Inc.
 Standard Oil Development Co.
 Udyllite Corp.
 Union Carbide & Carbon Research
 Labs., Inc.
 U. S. Steel Corp.

Vanadium Corp. of America
 Westinghouse Electric Corp.
 Youngstown Sheet and Tube Co.

It is hoped that this list of contributors will continue to grow so that an ultimate goal of at least \$100,000 will be assured.

There may be a number of companies which are not aware of this exposure test site program of the Society or if so have not received a general letter of invitation. In any event, if a company or organization is interested in supporting the program or in securing reprints of the article which appeared

in the October ASTM BULLETIN, it would be appreciated if they would indicate their interest to ASTM Headquarters.

There has been some question in the past as to whether ASTM test site facilities are available to organizations other than ASTM committees. The answer to this question is "yes," and it is planned that the rules and regulations regarding such applications will be published in a forthcoming issue of the BULLETIN as well as other pertinent information regarding the status of the test sites.

Joint Committee on Effect of Temperature to Underwrite Extensive Research Programs

ASTM-ASME Committee Has Important Strategic Programs Involving Materials for Aircraft, Gas Turbines, Power Generating Equipment, and Cast Iron

REPORTS from the several panels of the Joint ASTM-ASME Committee on the Effect of Temperature on the Properties of Metals during the committee's several sessions in New York, November 27-30, inclusive, indicated no diminution in the committee's far-flung technical activities, including several important research projects. Several of these involve critical materials and the committee is expediting this work. A Finance Committee under the chairmanship of N. L. Mochel, with the close collaboration of the Joint Committee officers, Ernest L. Robinson, General Electric Co., and Howard Cross, Battelle Memorial Inst., is undertaking fund-raising work which it is hoped will reach \$100,000, to underwrite adequately the important projects either already under way or to be started soon.

This joint committee has had a particularly notable record of achievements down through the years, and the researches it has carried through and the numerous publications it has sponsored have provided American industry with a wealth of important information on the properties of metals at both high and low temperatures. For the past two years the work has gone on at an increasing tempo. For example, at the ASTM Annual Meeting at Atlantic City in June, 1950, some six separate sessions were devoted to reports and papers in this field, including a Symposium on the Effect of Sigma Phase on the High-Temperature Properties of Metals (see p. 20, this BULLETIN), Symposium on Corrosion of Gas Turbine Materials, round-table discussions on problems with turbine forgings, and several miscellaneous papers including three on light metals. These sym-

posiums are in course of publication.

Last spring the committee sponsored the publication of a notable Report on the Strength of Wrought Steels at Elevated Temperatures, prepared by Messrs. R. Miller and J. J. Heger of the Carnegie-Illinois Steel Corp. This report, with its extensive data on the physical properties, including the creep and creep-rupture strengths of both carbon and alloy steels, went a long way toward meeting the critical demands for current information on the properties of metals that are being extensively used in power plant and related work. Among the current projects is the compiling of more extensive data on a wide range of metals.

While considerable could be written about each of the research projects which the committee either has under way or in the planning stage, with materials actually being prepared and collected by the committee, space at this stage permits just a listing of the formal projects:

- A. Statistical Study of the High-Temperature Properties of Sheet Materials. Considerable work already under way; materials being gathered.
- B. Extensive Solicitation and Compilation of Data on High-Temperature Properties of Metals. First work on heat-resisting steels well under way.
- C. Work on the Fatigue Properties of Materials Going into the Gas Turbine. Program outlined.
- D. Elevated Temperature Tests on Cast Iron—to Be Directed by a Committee of Past and Present Chairmen of ASTM Committee A-3, functioning under

the Steam Power Panel of the Joint Committee.

- E. A Fundamental Study of the Actual Properties of a Basic ASTM Steel Plate Specification Material and a Basic Pipe Specification Material. This work is of vital interest to certain stress establishing bodies.
- F. Work in Low-Temperature Field:
 1. To examine procedures for low-temperature testing, in order to (a) correlate test procedures with service results and (b) formulate new test procedures where desirable.
 2. To summarize available information on the properties of materials at low temperatures, in order to (a) correlate this information with service failures and (b) sponsor the development of new materials where desirable.
 3. To examine present procedures for bonding (soldering, brazing, and welding) of low-temperature materials, in order to (a) correlate laboratory tests with service experience and (b) develop new bonding techniques where desirable.

In the next few months the committee will publish the first section of a continuing report on up-to-date high-temperature properties of materials, particularly the creep and creep-rupture properties. The results of an extensive questionnaire on the alloys of iron and chromium and nickel are now being compiled. The current materials situation will make these data of great value. Already under way is the solicitation from leading American and foreign concerns of similar data on the "super alloys," and another important segment of materials used at elevated temperatures, namely the ferritic alloys, including chromium and chromium-molybdenum steels.

Changes in Concrete Pipe Requirements

A good representation of producer and consumer members applied themselves diligently to an impressive agenda at a two-day meeting of Committee C-13 on Concrete Pipe held on December 7 and 8 at the Statler Hotel, St. Louis, Mo. A number of proposed revisions of existing standards were approved which when adopted will materially improve their interpretation and use. These include changes in the description and application of the upper bearing block in the three-edge-bearing method allowing an extension over the bell to utilize the full length of the pipe and also to provide for the distribution of the load through a rigid steel member; the addition of a section in the Specification for Non-Reinforced Sewer Pipe (C 14) to permit the use of tongue-and-groove pipe; and the elimination of inconsistencies between the three sewer pipe specifications (C 14, C 75, and C 76) by specifying minimum strength requirements and including a section to cover acceptance or rejection on a strength basis where not already provided.

Further tentative revisions will provide a requirement that welds shall develop 75 per cent of the minimum strength of the wire, or 52,500 psi., in both reinforced-concrete pipe specifications (C 75 and C 76); include the use of

all portland cements meeting the requirements of current ASTM Specifications for Portland Cement (C 150), Air-Entraining Portland Cement (C 175), and Portland Blast-Furnace Slag Cement (C 205); permit the use of admixtures or blends if acceptable to the consumer; change the permissible cover over steel in C 75 and C 76 to apply to reinforcement in the barrel of the pipe requiring not less than $\frac{3}{4}$ in. at any point; and delete the maximum size requirement of coarse aggregate in the Specification for Concrete Irrigation Pipe (C 118).

A significant revision adopted was the substitution of a "fill" or permeability test for the existing hydrostatic test as referred to in Section 9 of Specifications C 14. It is felt that the present hydrostatic test is impractical and unnecessary for this type of pipe.

Consideration was given to suggestions from the Technical Problems Committee of the American Concrete Pipe Assn. The recommendation that compressive strength requirements on the concrete only on pipe larger than 72 in. in diameter and that the three-edge bearing test be used only on pipe 72 in. in diameter or smaller was referred to a subcommittee for further study. The proposal that the ultimate failure test be discontinued is to be studied, there

being some hesitancy on taking immediate action due to the possible significance of information on the behavior of pipe between the 1/100-in. crack and the ultimate failure points. A proposed revision of Specifications C 75 with corresponding change in Specifications C 76, permitting the use of cored cylinders for determining strength requirements in the large-size pipes was referred to a committee for further consideration. A special subcommittee was appointed to prepare a recommended practice on bedding and back filling.

Considerable discussion took place on a proposed specification for reinforced concrete low-head pressure pipe for sewer purposes. Action was taken to develop the proposed specifications at an early date.

A series of compressive strength tests on reinforced-concrete pipe by the three-edge bearing method was suggested to check theoretical method of design, relate strength tests to minimum design requirements, and to obtain information regarding strength and design requirements for large-diameter pipe. Secretary Peckworth was instructed to prepare and circulate a letter to all producers and possibly state highway departments soliciting all available data to assist in the research work of the committee.

November, 1951 was suggested as a tentative time for the next meeting of the committee.

New ASTM Committee on Radioactive Isotopes

MENTION has been made in the ASTM BULLETIN of the organization of a new ASTM Committee on Radioactive Isotopes. The personnel of this new committee, to be designated E-10, has been assembled as set forth below and it is expected that an organization meeting will be held shortly. Dr. George G. Manov of the U. S. Atomic Energy Commission is serving as temporary chairman to effect organization.

The purpose of the committee is to serve in an advisory capacity to the various ASTM technical committees that may be interested in test procedures involving the use of radioactive isotopes. The committee will also concern itself with the fundamental considerations in such test procedures.

PERSONNEL OF NEW ASTM COMMITTEE ON RADIOACTIVE ISOTOPES

E. B. Ashcraft, Westinghouse Electric Corp.
A. Allan Bates, Portland Cement Assn.
G. D. Calkins, Battelle Memorial Inst.
S. Edward Eaton, Arthur D. Little, Inc.
A. C. Fieldner, U. S. Bureau of Mines
C. D. Foulke, Weirton Steel Co.
W. H. Fulweiler, Consulting Chemist
J. W. Garrison, Armour Research Foundation
E. D. Haller, Beckman Instrument Co.
W. N. Harrison, National Bureau of Standards
H. E. Hosticka, U. S. Bureau of Reclamation
J. W. Irvine, Jr., Massachusetts Institute of Technology
J. L. Kuranz, Nuclear Instrument and Chemical Corp.
S. K. Love, U. S. Geological Survey

George G. Manov (Temporary Chairman), U. S. Atomic Energy Commission
L. J. Markwardt, U. S. Forest Products Lab.
D. M. McCutcheon, Ford Motor Co.
Homer S. Myers, Radioactive Products, Inc.
J. H. Phillips, Babcock and Wilcox Co.
R. G. Russell, Gulf Research and Development Co.
Wm. B. Snow, Kellogg Corp.
Orville J. Sweeting, University of Colorado
L. S. Taylor, National Bureau of Standards
T. Smith Taylor, U. S. Testing Co.
Ernest H. Wakefield, Radiation Counter Lab.
C. E. Weber, General Electric Co.
J. R. White, Socony-Vacuum Labs.
J. Norton Wilson, Shell Development Co.
L. A. Wooten, Bell Telephone Labs.
W. H. Yando, Monsanto Chemical Co.

First ISO Meeting in USA

THE ISO Technical Committee on Rubber, ISO/TC45 met at Akron, Ohio, October 16-20, the first technical committee of the International Organization for Standardization to hold a meeting in the United States. Representatives from France; Italy, The Netherlands, New Zealand, Switzerland, United Kingdom, and USA were present.

Discussions during the meeting centered largely on the following subjects connected with rubber technology: Hardness; tension stress-strain; tear strength; ply adhesion; aging; abrasion; grading of raw rubber; inter-laboratory variations; mechanical stability and viscosity of latex; flex-cracking; rubber-to-metal-bonding; classification of vulcanized rubber.

Standardization of rubber test methods and products is valuable because of the international character of the rubber industry, in the sense that the raw materials are generally not produced in the country where they are fabricated, and fabricated rubber products are a big item in the export trade of the most important industrial countries.

General arrangements for the meeting were handled by a committee of rubber manufacturers and users under the chairmanship of L. V. Cooper, Firestone Tire and Rubber Co. The officers of ASTM Committee D-11 on Rubber and Rubber-like Materials, namely, Simon Collier, Chairman, and Arthur W. Carpenter, Secretary, took an active part in making these arrangements. It was on the invitation of the committee that the meeting was held in America, and arrangements were made for the participation on the part of a number of active D-11 members. Executive Secretary C. L. Warwick welcomed the foreign delegates to this first meeting of an ISO committee to be held in America.

1951 Annual Meeting Forest Products Research Society

NATIONAL meeting of unusual interest to a large portion of the Society membership will be held in Philadelphia next May by the Forest Products Research Society. This meet-

ing will be of special interest for two reasons. From the standpoint of those directly or indirectly connected with the forest products industry, the program, running from May 7 to 11, will present a series of seven technical sessions and a novel exhibit of equipment and wood products. The second reason is that one of the technical sessions will be co-sponsored by ASTM, through Committee D-7 on Wood.

The technical sessions consisting of from one to six papers each will be co-sponsored, in addition to ASTM, by other national societies including the Society of American Foresters, Wood Industries Division of the ASME, TAPPI, Northeastern Utilization Council, and Wood Adhesives Division, ASPI. Arrangements for the ASTM sponsored session have already been completed with President Markwardt, Chairman of Committee D-7.

The exhibit, to be known as the International Industry Show, will consist of inside exhibits in Convention Hall, as well as a Logging and Sawmill Show to be held in an outdoor area. More detailed information will be published in a succeeding issue of the BULLETIN covering these programs.

Descriptions of Constituents of Natural Mineral Aggregates

EDITOR'S NOTE.—This compilation has been prepared by Subcommittee II-f on Aggregate Mineralogical Characteristics as Related to Concrete of Committee C-9 on Concrete and Concrete Aggregates, under the chairmanship of D. O. Woolf, U. S. Bureau of Public Roads. These descriptions are being published as information only in order to solicit further comments preliminary to further consideration for adoption by the Society. Comments should be addressed to the Society, 1916 Race St., Phila. 3, Pa.

SCOPE

The purpose of this compilation is to provide brief, useful, and accurate descriptions of some of the more common or more important natural materials found as constituents of mineral aggregates. The descriptions have been prepared to provide a basis for understanding these terms when they are used to designate aggregate constituents. It should be emphasized that many of the materials described frequently occur in particles that do not display all of the characteristics given in the descriptions and that most of these materials grade from varieties meeting one description to varieties meeting another, with all intermediate stages being found.

These descriptions are not adequate to permit the identification of materials, since the accurate identification of the natural constituents of mineral aggregates can, in many cases, only be made by a qualified geologist, mineralogist, or petrographer using the apparatus and procedures of these sciences. Reference to these descriptions may, however, serve to indicate or prevent gross errors in identification. Identification of the constituent ma-

terials in a mineral aggregate may assist in recognizing its properties, but identification alone, however accurately it may be accomplished, cannot provide a basis for predicting the behavior of aggregates in service. Mineral aggregates composed of constituents of any type or combination of types may perform well or poorly in service depending upon the exposure to which they are subjected, the physical and chemical properties of the matrix in which they may be embedded, their physical condition at the time they are used, and upon other factors.

INTRODUCTION

The natural materials found as constituents of mineral aggregates are, for the most part, particles of rocks and minerals. Rocks are classified according to origin into three major groups: igneous, sedimentary, and metamorphic; and are subdivided into types according to mineral and chemical composition, texture, and structure. Most rock particles are composed of mineral grains of more than one type. However, in some cases a rock may be composed of grains of only one mineral.

Certain examples of the rock quartzite are composed exclusively of the mineral quartz. The particles composing the finer sizes of many sands frequently consist of individual mineral grains. Descriptions are therefore given not only of rock types but also of minerals.

DESCRIPTIONS OF MINERALS

For the purpose of indicating significant relationships, the descriptions of minerals are presented in groups as follows:

(a) Silica Minerals:

(1) *Quartz*.—Quartz is a hard mineral (will scratch glass and not be scratched by a knife) composed wholly of silica (silicon dioxide). When pure it is colorless with a glassy (vitreous) luster and a shell-like (conchoidal) fracture. It lacks a visible cleavage, and, when present in massive rocks such as granite, it usually has no characteristic shape.

(2) *Opal*.—Opal is a hydrous form of silica which occurs as an amorphous mineral and therefore is without characteristic external shape or internal crystalline arrangement. It has a variable water content ranging from 2 to 10 per cent. The specific gravity and hardness are always less than those of quartz. The color is variable and the luster is resinous to glassy. It is usually found in sedimentary rocks and is the principal constituent of diatomite, but it is also found as a secondary

material filling cavities and fissures in igneous rocks. It is of particular importance as a constituent of mineral aggregates because of its reactivity with the alkalis in portland cement.

(3) *Chalcedony*.—Chalcedony has been considered both as a distinct mineral and as a variety of quartz. It is now generally believed to be composed of a submicroscopic mixture of fibrous quartz with a smaller but variable amount of opal. The properties of chalcedony are intermediate between those of opal and quartz, from which it can be distinguished only by laboratory tests. It frequently occurs as a constituent of the rock chert and is reactive with the alkalis in portland cement.

(4) *Tridymite and Cristobalite*.—These minerals are crystalline forms of silica which are sometimes found in volcanic igneous rocks. They are metastable at ordinary temperatures and pressures. Unless they occur in well-shaped crystals they can only be distinguished from quartz by laboratory tests. They are rare minerals and are included here only because of their reactivity with cement alkalis.

(b) *Feldspars*.—The minerals of the feldspar group are the most abundant rock-forming minerals. They are silicates of aluminum, and, since they all have good cleavage in two directions, particles of feldspar usually show several smooth surfaces. Frequently, the smooth cleavage surfaces show fine parallel lines. All feldspars are softer than, and can be scratched by, quartz. The various members of the group are differentiated by chemical composition and crystallographic properties. The potash feldspars are *orthoclase* and *microcline*. The *plagioclase* group includes those with soda or lime or both and forms a continuous series including *albite*, *oligoclase*, *andesine*, *labradorite*, *bytownite*, and *anorthite*. The soda and potash feldspars occur typically in granitic rocks, while those of increasing lime content are found in rocks of decreasing silica content such as diorite, gabbro, and basalt.

(c) *Micaceous Minerals*.—The micaceous minerals characteristically have a perfect cleavage. Particles of such minerals can therefore usually be split into extremely thin flakes. The true micas are usually colorless or light green (*muscovite*); or dark green, dark brown, to black (*biotite*), and have elastic flakes. The green micaceous material often found in schists usually represents minerals of the *chlorite* group which may be distinguished from the micas because they form comparatively non-elastic flakes.

(d) *Carbonate Minerals*.—The most common carbonate mineral is *calcite* (calcium carbonate). The mineral *dolomite* consists of calcium carbonate and magnesium carbonate in equivalent chemical amounts, which are 54.27 and 45.73 per cent by weight, respectively. Pure limestones are composed principally of calcite; magnesian limestones contain both calcite and dolomite. Both calcite and dolomite are relatively soft, the hardness of calcite being 3 and that of dolomite $3\frac{1}{2}$ to 4 on the Mohs scale, and are readily scratched by a knife blade. They have rhombohedral cleavage

which results in their breaking into fragments with smooth parallelogram-shaped sides. Calcite is soluble with effervescence in cold dilute hydrochloric acid; dolomite is soluble only if the acid is heated or if the sample is powdered.

(e) *Ferromagnesian Minerals*.—The various types of igneous rocks contain characteristic dark green to black minerals. These are generally silicates of iron or magnesium or both and include the minerals of the *amphibole* and *pyroxene* groups. The most common amphibole is *hornblende*; the most common pyroxene is *augite*. Black mica, *biotite*, may also be considered as a ferromagnesian mineral. Amphiboles, pyroxenes, and biotite may also be found in marble. *Olivine*, usually olive-green in color, is a characteristic mineral of igneous rocks of very low silica content.

(f) *Clay Minerals*.—The term "clay" refers to a rock composed of particles of a specific size range, and containing appreciable quantities of clay minerals (hydrous aluminum silicates). Clay minerals are formed by the alteration of feldspars, other silicate minerals, and volcanic glass. Most particles consisting of clay minerals are soft and porous, and some clay minerals of the *montmorillonite* and *illite* (hydromica) groups (swelling clays) undergo large volume changes with wetting and drying. Clay minerals are found in seams and pockets of limestones, in weathered igneous rocks, and are important constituents of shales.

(g) *Sulfides*.—Many sulfide minerals are important ores of metals, but only *pyrite* and *marcasite*, both sulfides of iron, are frequently found in mineral aggregates. Pyrite is found in igneous, sedimentary, and metamorphic rocks; *marcasite* is much less common and is found only in sedimentary rocks. Pyrite is brass yellow in color and has a metallic luster; *marcasite* is also metallic but lighter in color. Pyrite is often found in cubic crystals. *Marcasite* often oxidizes with the liberation of sulfuric acid; pyrite does so less readily. Both minerals are known as "fool's gold."

(h) *Iron Oxides*.—The common iron oxide minerals may be grouped in three classes: (1) Black, magnetic: *magnetite*; (2) Red or reddish when powdered: *hematite*; (3) Brown: *limonite*. Magnetite is an important accessory mineral in many dark igneous rocks. Limonite is a term applied loosely to a variety of brown minerals some of which are hydrous and include the iron minerals in many ferruginous sandstones, shales, and clay ironstones.

(i) *Zeolites*.—The zeolite minerals comprise a large group of soft, hydrous silicates usually white or light colored, formed as a secondary filling in cavities or fissures in rocks. Some zeolites, particularly *laumontite* and *heulandite*, are reported to be reactive with cement alkalis.

DESCRIPTIONS OF IGNEOUS ROCKS

Igneous rocks are those that have been formed by cooling from a molten mass. They may be divided into two classes: (1) *Coarse Grained* (Intrusive, deep-seated), and (2) *Fine Grained* (Extrusive, surface,

volcanic) rocks. The coarse-grained, intrusive rocks cooled slowly within the earth. The fine-grained, extrusive rocks formed as rather quickly cooled lavas and frequently contain natural glass. The *porphyries* are characterized by the presence of large mineral grains in a fine-grained ground mass. This texture is the result of a sharp change in the rate of cooling during the solidification of the rock.

Within the two classes, rocks are usually classified and named on the basis of their mineral content, which in turn depends to a large extent on the chemical composition. Rocks in the intrusive class generally have chemical equivalents in the extrusive class.

Coarse-Grained Intrusive Igneous Rocks:

(1) *Granite*.—Granite is a medium- to coarse-grained, light-colored rock characterized by the presence of quartz and feldspar. The feldspars are potash feldspars (orthoclase or microcline or both) or soda plagioclase or both. Feldspar is usually more abundant than quartz. Dark-colored mica (*biotite*) is usually present and light-colored mica (*muscovite*) frequently. Other dark-colored minerals, especially hornblende, may be present in amounts less than those of the light colored constituents. *Quartz-monzonite* and *granodiorite* may be mentioned as rocks similar to granite, but containing more plagioclase feldspar.

(2) *Syenite*.—Syenite is a medium- to coarse-grained, light-colored rock composed essentially of potash feldspar, generally orthoclase. Quartz is generally absent. Dark ferromagnesian minerals such as hornblende, biotite, or pyroxene may be present.

(3) *Diorite*.—Diorite is a medium- to coarse-grained rock composed essentially of plagioclase feldspar and one or more ferromagnesian minerals such as biotite, hornblende, or pyroxene. The plagioclase is intermediate in composition, usually of the variety known as andesine. Diorite is darker in color than granite or syenite and lighter than gabbro. If quartz is present, the rock is called *quartz diorite*.

(4) *Gabbro*.—Gabbro is a medium- to coarse-grained, dark-colored rock consisting essentially of ferromagnesian minerals and plagioclase feldspar. The ferromagnesian minerals may be pyroxenes, amphiboles, or both. The plagioclase is one of the lime varieties such as labradorite. Ferromagnesian minerals are usually more abundant than feldspar. *Diabase* is rock of similar composition to gabbro and basalt but is intermediate in mode of origin, usually occurring in smaller intrusions than gabbro, and having a medium grained texture. The term "trap" or "trap rock" is a collective term for dark-colored, fine- to medium-grained igneous rocks such as diabase and basalt.

(5) *Pyroxenite and Peridotite*.—Rocks composed almost entirely of pyroxene or olivine are called pyroxenites or peridotites.

Rocks of these types are relatively rare but their metamorphosed equivalent, serpentinite, is more common.

(6) *Pegmatite*.—Extremely coarse-grained varieties of igneous rocks are known as pegmatites. These are usually light colored and are generally equivalent to granite or syenite.

Fine Grained Extrusive Igneous Rocks:

The fine-grained equivalents of the coarse-grained igneous rocks described above have similar chemical compositions. The extrusive rocks are so fine-grained that the individual mineral grains are usually not visible to the naked eye. They may contain the same constituent minerals, or these minerals may be replaced in part or wholly by natural glass.

(1) *Obsidian, Pumice, and Perlite*.—Igneous rocks composed wholly of glass have been named on the basis of their texture. A dense natural glass is called obsidian, while a glassy froth filled with bubbles is called pumice. A siliceous or glassy lava with an onion-type structure and a pearly luster, containing 2 to 5 per cent water, is called perlite. When heated quickly to the softening temperature, perlite puffs to become a synthetic pumice. These rocks may be reactive with the alkali in portland cement.

(2) *Felsite*.—Light-colored, fine-grained extrusive rocks are collectively known as felsite. The felsite group includes *rhyolite*, *dacite*, *andesite*, and *trachyte* which are the extrusive equivalents of granite, quartz diorite, diorite, and syenite, respectively. These rocks are usually light colored but may be dark red or even black at times. When they are dark they are more properly classed as "trap" (see Gabbro). When they contain natural glass, the glass frequently has such a high silica content that it is reactive with cement alkalis.

(3) *Basalt*.—Basalt is the fine-grained extrusive equivalent of gabbro. When basalt contains natural glass, the glass is generally lower in silica content than that of the lighter-colored extrusive rocks and is hence less likely to be reactive with cement alkalis.

DESCRIPTIONS OF SEDIMENTARY ROCKS AND THEIR METAMORPHIC EQUIVALENTS

Sedimentary rocks are stratified rocks laid down for the most part under water, although in some cases wind action may have been important. They may be composed of particles of pre-existing rocks de-

rived by mechanical agencies or they may be of chemical or organic origin.

(a) *Carbonate Rocks*.—The common carbonate rocks are *limestones* and *magnesian limestones* ("dolomites"). The purer limestones are composed principally of grains of calcite; magnesian limestones contain calcite and dolomite. A magnesian limestone composed almost entirely of the mineral dolomite may be given the rock name "dolomite." Most limestones contain some noncarbonate impurities such as silica, clay, organic matter, or hydrous calcium sulfate (gypsum). When considerable sand is present, the rock becomes a sandy (or "arenaceous") limestone; if shale or clay minerals are abundant it becomes a clayey (or "argillaceous") limestone. A metamorphosed limestone after recrystallization is known as a *marble*. Very soft carbonate rocks are known as *chalk*, *marl*, or "*lime rock*."

(b) *Conglomerates, Sandstones, and Quartzites*.—These rocks consist of particles of sand or gravel or both cemented together. If the particles include a considerable proportion larger than sand, the rock is a conglomerate. If the particles are in the sand sizes, the rock is a sandstone or a quartzite. If the rock, when fractured, breaks around the sand grains, it is a sandstone; if the fracture passes through the grains it is a quartzite. Conglomerates and sandstones are sedimentary rocks. Quartzites may be sedimentary or may be metamorphosed sandstones. The cementing material may be quartz, opal, calcite, dolomite, clay, iron oxides, or other materials. If the nature of the cementing material is known, the designation of the rock may include a reference thereto, as "*opal-bonded sandstone*," or "*ferruginous conglomerate*."

Graywacke is sandstone in which many of the sand grains are dark in color and which usually contains fairly large amounts of cementing material resembling shale or slate.

Arkose is coarse-grained sandstone containing conspicuous amounts of feldspar and is derived from granite.

(c) *Argillaceous Rocks*.—These rocks are largely composed of or derived from sedimentary silts and clays. When relatively soft they are known as *claystones*, or *siltstones*, depending on the particles of which they are composed. When harder they are known as *shales*, and when metamorphosed they become, with progres-

sively greater alteration, *slates*, *phyllites*, and *schists*. All of these rocks are usually characterized by a laminated structure and a tendency to break into thin particles.

(d) *Chert*.—Chert is a fine-grained rock which is characterized by hardness (scratches glass, is not scratched by a knife blade), conchoidal (shell-like) fracture in dense varieties, the fracture becoming splintery in porous varieties, and a variety of colors. The dense varieties are very tough and are usually gray to black, or white to brown in color, less frequently green, red, or blue, and have a waxy to greasy luster. The porous varieties are usually lighter in color, most frequently being white or stained yellowish, brownish, or reddish, and have a chalky surface. Dense red and, in some cases, dense yellow, brown, or black chert is sometimes called "*jasper*." Dense black and, in some cases, dense gray chert is sometimes called "*flint*." Chert is composed of silica in the form of chalcedony, cryptocrystalline quartz, or opal, or combinations of any of these three. The determination of which form or forms of silica are present requires careful determination of optical properties, absolute specific gravity, or both. Chert occurs most frequently as nodules or bands in limestones and in sands and gravels derived from such rocks.

DESCRIPTIONS OF METAMORPHIC ROCKS

Since the typical metamorphic equivalents of sedimentary rocks have been described under Sedimentary Rocks, the descriptions below cover metamorphosed igneous rocks:

(a) *Serpentine*.—Serpentine is a relatively soft light to dark green rock formed usually from igneous rocks such as pyroxenites and peridotites. It may contain some of the original pyroxene or olivine but is largely composed of softer hydrous minerals. Very soft talc-like material is often present in serpentine.

(b) *Gneiss*.—Gneiss is usually formed by the metamorphism of schists or igneous rocks. It is characterized by a layered structure resulting from approximately parallel lenses and bands of platy minerals, usually micas. Gneisses are usually coarser grained than schists and usually contain an abundance of feldspar. All intermediate varieties between gneiss and schist and between gneiss and granite are found, often in the same areas in which well-defined gneisses occur.

Measurement of Density of Hydrocarbon Liquids by the Pycnometer—An Abstract¹

By H. M. Smith² and Cooperators³

¹ Complete paper with the same title and by the same author appeared in *Analytical Chemistry*, Vol. 22, November, 1950.

² Bureau of Mines, Bartlesville, Okla.
³ R. C. Alden, Phillips Petroleum Co., E. L. Baldeschwieler, Standard Oil Development Co., G. R. Bond, Jr., Houdry Process Corp., L. M. Henderson, Pure Oil Co., S. S. Kurtz, Jr., Sun Oil Co., L. Lykken, Shell Development Co., H. Levin, Texas Co., R. Matteson, California Research Corp., E. T. Scafe, Socony-Vacuum Oil Co., C. E. Starr, Jr., Esso Standard Oil Co. (La.), M. J. Stross, Universal Oil Products Co.

SECTION D of Research Division IV on Hydrocarbon Analysis has cooperatively tested the ASTM Standard Method of Measurement of Density of Hydrocarbon Liquids by the Pycnometer, D 941 - 49, using a 5-ml. pycnometer with isopentane, a 12-lb. Reid vapor pressure gasoline, and toluene. The results substantiate the claims reported by Lipkin *et al.*⁴ that the aver-

⁴ M. R. Lipkin, J. A. Davison, W. T. Harvey, and S. S. Kurtz, Jr., *Industrial and Engineering Chemistry, Anal. Ed.*, Vol. 16, No. 1, p. 55 (1944).

age deviation is ± 0.0001 g. per ml. For example, out of thirty-five individual determinations on the 12-lb. Reid vapor pressure gasoline, thirty deviated from the laboratory average by either 1 or 0 in the fourth decimal place, four had a deviation of 2, and in only one was a deviation of 3 in the fourth decimal place reported.

Cooperative data using the Bingham pycnometer or a five place chainomatic balance indicate similar precision and accuracy.

PERSONALS...

News items concerning the activities of our members will be welcomed for inclusion in this column.

NOTE—These "Personals" are arranged in order of alphabetical sequence of the names. Frequently two or more members may be referred to in the same note, in which case the first one named is used as a key letter. It is believed that this arrangement will facilitate reference to the news about members.

Alan R. Albright has retired as Associate Technical Director of Best Foods, Inc., Indianapolis. He had been with the company since 1936.

Milton R. Beasley, formerly Consultant on Asphalt and Asphalt Roofing, Norwood, Mass., is now Assistant to the President, Central Commercial Co., Chicago.

Robert D. Bonney, formerly Assistant Manager of Manufacturing, has been appointed Manager of Manufacturing at Congoleum-Nairn, Inc., Kearny, N. J. Mr. Bonney is the recently elected President of the Federation of Paint and Varnish Production Clubs. He is very active in ASTM and has given service in many technical and administrative activities. At present he is an ASTM Director.

Arthur E. Cozens is now associated with the Pacific Scientific Co., Los Angeles, Calif., as Sales Engineer. He was previously Material & Process Engineer, U. S. Marine Corps Air Station at El Toro.

V. M. Darsey has been made Vice-Chairman of the Board of Directors of the Parker Rust-Proof Co., Detroit, Mich.

For some time he has been President of the organization. Mr. Darsey is active in various technical phases of ASTM work and is a Past-Chairman of the Detroit District Council. **F. J. DeWitt**, formerly Assistant Sales Manager of the Parker Company, has been appointed a Vice-President.

J. Emil Engstrom is now Quality Control Engineer, Marlin-Rockwell Corp., Jamestown, N. Y. He was previously associated with the Arcos Corp., Philadelphia, Pa., in a similar capacity.

Arthur Eldridge Focke, Chief Metallurgist of the Diamond Chain Co., Indianapolis, has been appointed head of the Chemistry and Metallurgy Department of the Fairchild Engine and Airplane Corp.'s NEPA division, Oak Ridge, Tenn.

William A. Hamor, Assistant Director of Mellon Institute, was named by the Pittsburgh Section of the American Chemical Society to receive its Pittsburgh Award for the year 1950 for outstanding service to chemistry, the presentation being made at a dinner in the University Club in December.

Stewart H. McGaw, a recent graduate

of Stockton Junior College, Stockton, Calif., is now associated with Standard Materials, Inc., Modesto, Calif.

Charles Pack, formerly Vice-President in Charge of Production, was recently elected Vice-President in charge of the newly formed Engineering and Research Department of Doehler-Jarvis Corp., New York City.

L. B. Parsons recently was promoted to Assistant Director of Research in the Research and Development Department of Lever Brothers Co., Cambridge, Mass.

Willis J. Porter, formerly Materials and Concrete Engineer, Perkins Gravel Co., Sacramento, Calif., is now associated with O. J. Porter & Co., of the same city, in a similar capacity.

James I. Schwartz, until recently Research Associate, National Bureau of Standards, Washington, D. C., is now Plant Engineer, National Brick & Supply Co., Inc., Terra Cotta, D. C.

Konrad Stern, formerly Senior Field Engineer, Barber Oil Corp., New York City, is now with Brighton Terminal Ltd., Trinidad, B. W. I., in a similar capacity.

Gilbert Thiessen, Technical Adviser for the Chemical Division of Koppers Co., Inc., Pittsburgh, Pa., has been named Manager of the Laboratory Section of the company's Central Research Department, and will supervise and direct all Koppers laboratory research performed in company-owned laboratories or plants and by contract in academic and commercial laboratories.

Lincoln T. Work, Consulting Engineer, New York City, has been elected a Director of the Association of Consulting Chemists and Chemical Engineers of that city, to serve for a three-year term.

NEW MEMBERS...

The following 16 members were elected from November 9, to December 20, 1950, making the total membership 6826... Welcome to ASTM

Note—Names are arranged alphabetically—company members first then individuals

Chicago District

ACME ALUMINUM FOUNDRY CO., C. G. Sidinger, Chief Engineer, 6831 S. Bell Ave., Chicago 36, Ill.

MILWAUKEE BOILER MANUFACTURING CO., E. J. Berry, Chief Engineer, 1101 S. Forty-first St., Milwaukee 46, Wis.

FENTRESS, D. WENDELL, Vice-President, Engineering, Chicago Metal Hose Corp., 840 N. State St., Elgin, Ill.

SIMONS, ALLISON K., Research Engineer, Bostrom Manufacturing Co., 133 W. Oregon St., Milwaukee 4, Wis.

Cleveland District

PERRINE, R. J., Metallurgist, The Electric Furnace Co., Wilson St. and PRR, Salem, Ohio. For mail: 427 N. Main, Columbiana, Ohio.

New England District

TOWNSEND, HENRY B., Vice-President, General Latex and Chemical Corp., 666 Main St., Cambridge 39, Mass.

New York District

HOLLAND, JOHN MICHAEL, Assistant to Director of Research, Pepperell Manufacturing Co., 40 Worth St., New York, N. Y.

MIKOCHIK, STEPHEN, Instructor, Civil Engineering Dept., Manhattan College, Bronx 63, N. Y. [J]*

Philadelphia District

KNOFF, OTTO O., Chief Chemist, Federated Metals Division, American Smelting and Refining Co., Hedley and Richmond Sts., Philadelphia, Pa. For mail: Haldeman

Ave. and Red Lion Rd., Philadelphia 15, Pa.

WILENCHIK, I. W., President, Metallurgical Products Co., Thirty-fifth and Moore Sts., Philadelphia 45, Pa. For mail: 5612 Wynnefield Ave., Philadelphia 31, Pa.

Southern California District

KEYSTONE ENGINEERING CO., Lester A. Olson, Chief Engineer, 1444 S. San Pedro St., Los Angeles 15, Calif.

SUMNER, EDWIN V., Metallurgist, Federated Metals Division, American Smelting and Refining Co., 4010 E. Twenty-sixth St., Los Angeles 23, Calif.

U. S. and Possessions

STILLIE, MELFRED JAMES, Field Representative, Dunham Concrete Co., Inc., 3000 N. Highland Ave., Albuquerque, N. Mex. For mail: 444 Loma Hermosa Dr., Albuquerque, N. Mex.

Other than U. S. Possessions

LABORATORIO PROVE MATERIALI POLITECNICO, P. Vacchelli, Professor, Piazza Leonardo da Vinci 32, Milan, Italy.

INDIA, THE MASTER GENERAL OF THE ORDINANCE, M.G.O. Branch (M.E. Directorate/M.G.O. Publications Section), Army Headquarters, New Delhi, India.

McCALLUM, NEIL M., Chief Engineer, Department of Public Works, Government of British Columbia, Parliament Bldgs., Victoria, B. C., Canada.

* [J] denotes Junior Member.

NECROLOGY...

The Death of the following members has been reported

H. E. CORMAN, Chief Chemist, Canada Packers, Ltd., Toronto, Canada (December, 1949). Member since 1947.

CARL A. HEUSSNER, Materials Engineer, Chrysler Corp., Detroit, Mich. (January 1, 1951). Member since 1933. (See accompanying article.)

GUY H. MERRY, Secretary, Merry Brothers Brick and Tile Co., Augusta, Ga. (July 3, 1950). Representative of sustaining membership of his company since 1938. A valuable man in the Merry Brothers Co., Mr. Merry died in Asheville, N. C., after suffering a series of heart attacks.

BERT E. SANDELL, Metallurgist and Chief Chemist, Stewart Die Casting Division of Stewart-Warner Corp., Chicago, Ill. (November 14, 1950). Member since 1937. Member of Committees B-6 on Die-Cast Metals and Alloys, and B-7 on Light Metals and Alloys, Cast and Wrought, since 1937, serving on subgroups of both committees; also since 1943 representative of Committee B-6 on Subcommittee on Spray Test of Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys. Mr. Sandell's entire experience was with the Stewart Corp. In addition to his sustained interest in ASTM technical work he was active in other societies and had served on the War Production Board.

Carl E. Heussner 1899-1951

IN THE death of Carl E. Heussner, Materials Engineer, Chrysler Corp., Detroit, Mich., on January 2, the Society loses a member who had been extremely active in various phases of its work and who had contributed a great deal to the technical, district, and administrative affairs of the Society. A member of ASTM since 1933, he had been concerned particularly with the work of such committees as B-6 on Die Cast Metals and Alloys, B-3 on Corrosion of Non-Ferrous Metals and Alloys, and B-8 on Electrodeposited Metallic Coatings. An outstanding authority in these fields, he had served on numerous subcommittees and was an officer of several of them. Other groups in which he was active were A-5 on Corrosion of Iron and Steel and C-14 on Glass. He served the Society on its Administrative Committee on Papers and Publications, and was an active member of the Detroit District Council for many years. As chairman of the finance committee in connection with the 1948 Annual Meeting in Detroit, Mr. Heussner did much to underwrite that very successful affair, particularly the social activities. He was active in the work of other bodies, and was particularly interested in the American Electroplater's Society. In this record of appreciation of his many services, the Society also extends its sympathy to his family.

SAMA Asks Recognition of Priorities for Apparatus Industry

THE scientific instrument industry recently issued a warning that an acute shortage of instruments and laboratory apparatus, which could bottleneck the entire national defense program, is not far distant.

"All companies in the industry are starting 1951 with large backlogs of unfilled orders ranging from two months to one year," Kenneth Andersen, executive vice-president of the Scientific Apparatus Makers Association, trade association of the industry, said recently.

"These backlogs are increasing daily," he said. "Few of the orders are DO rated, yet practically all of them are directly defense supporting. Instrument and apparatus companies have been unable to

get enough raw materials and components to maintain normal production under these conditions, much less increase their production to meet the orders which have doubled since the start of the war in Korea and the rearmament program.

"Sometime during 1951, depending upon how fast production for national defense gets under way, the demand for instruments and apparatus to control production, to conduct research, and as components of munitions is going to outstrip the supply and potential supply under existing conditions to such an extent that a major bottleneck will exist."

Andersen said there is some recognition of this in Washington and that some recognition has been given, particularly in the allocation of aluminum, but that the industry needs overall recognition on priorities if the bottleneck is to be avoided.

LABORATORY SUPPLIES...

Catalogs and Literature and Notes on New or Improved Apparatus

Note—This information is based on literature and statements from apparatus manufacturers and laboratory supply houses.

Catalogs and Literature

Analytical Service Uses Mass Spectrometer—The December issue of "CEC Recordings" describes an Analytical Service which is available to industry. Purity determinations are routine for the service, although samples of exhaust gases, stack gases, and smog have also been analyzed. An improved mass spectrometer manufactured by the Consolidated Engineering Corp., announced in the September, 1950, issue of "CEC Recordings," is used in these analyses. It is announced that a new plant is under construction which will provide the Analytical Service with space and facilities for a greater volume of work.

Volume 4, No. 4, "CEC Recordings," December, 1950, Consolidated Engineering Corp., 620 N. Lake Ave., Pasadena, Calif.

Hand Micrometer—A hand micrometer, expected to play an important part in the calibration of flat stock on the production, is described in a new brochure. The brochure states that the accuracy demanded by specifications set down by ASTM and TAPPI regulations have been precisely maintained.

E. J. Cady & Co., 134 N. La Salle St., Chicago 2, Ill.

Projection Microscope—A new bulletin describing the Vickers Projection Microscope is available from the R. Y. Ferner Company. The microscope described was introduced 25 years ago using basically the same design as is used today. The brochure states as a fundamental feature of the instrument that all operations of examination and photography can be carried out from one operating position.

The Vickers design has been constantly improved in the course of years. Stated as characteristics of the instrument are versatility, convenience in use, precision construction, and ruggedness.

The R. Y. Ferner Co., 110 Pleasant St., Boston 48 (Malden Station), Mass.

Cinemicrographic Focusing Unit—A bulletin describing the Cooke Cinemicrographic Focusing Unit is now available from the Lovins Engineering Co. The brochure describes the outstanding features of the instrument and states that it is for production of normal or delayed motion pictures of translucent objects on 16 or 35-mm. film. Construction features are described in detail.

Lovins Engineering Co., 8203 Cedar St., Silver Spring, Md.

Universal Testing Machine—Bulletin No. 40 of the Tinius Olsen Testing Machine Co. describes the new Super "L" type Hydraulic Universal Testing Machine. Separate sections of the bulletin are devoted to history of the development of the machine; loading system; indicating system; recording unit; and specifications. Also shown are testing tools for use with the Super "L" machine.

Bulletin No. 40, Tinius Olsen Testing Machine Co., Easton Road, Willow Grove, Pa.

Kjeldahl Equipment—Precision Scientific Co. has just printed a 16-page Kjeldahl Bulletin No. 555, describing the entire line of Kjeldahl equipment, which has recently been standardized. In past years Kjeldahl equipment, with the exception of a few small standard units, has been handled as specialized engineering jobs designed

for individual needs. In the new bulletin, standard Kjeldahl models range from 2-unit to 24-unit digestion and distillation, heated by gas or a wide choice of electrical heats. Also described are microdigestion units, as well as extraction and digestion racks. All possible heating methods and combinations of units are clearly spelled out, each listing is complete on a page.

Bulletin No. 555, Precision Scientific Co., 3737 W. Cortland St., Chicago 47, Ill.

Variable Speed Stirrers and Midget Pumps—The folder describing several sizes of variable speed stirrers and midget pumps manufactured by the Eastern Industries, Inc., is available from E. Machlett & Son. Stirrers in light duty, medium duty, and heavy duty models are described.

E. Machlett & Sons, 220 E. 23rd St., New York 10, N. Y.

Proving Rings—A new bulletin describing Morehouse proving rings is available. Separate sections of the bulletin are devoted to definition, design, tension, and compression type proving rings, uses, measuring apparatus, and capacities.

Morehouse Machine Co., 233 W. Market St., York, Pa.

Electro-Chemograph for Polarographic Analysis—To provide information about recently announced polarographic equipment adaptable to operation from a standard 115-volt a-c. supply circuit, Leeds & Northrup Co., designer and manufacturer of Electro-Chemograph, Type E, has just issued an illustrated 18-page publication. Copies of the new catalog are now available and will be sent upon request.

Catalog EM9-90 (1), Leeds & Northrup Co., 4934 Stenton Ave., Philadelphia 44, Pa.

Table Model Universal Testing Machine—Machines for tension, compression, flexure, shear, transverse tests are described in brochure 501 of National Forge and Ordnance Co. The brochure describes the medium-capacity unit-type universal testing machine which is designated as TMU and HMU. Features of the instrument are extensively described.

Brochure 501, National Forge and Ordnance Co., Irvine, Warren Co., Pa.

Dielectric Moisture Meter—A new 4-page illustrated bulletin describes TAG's improved Dielectric Moisture Meter. The bulletin, No. 1263, explains details of construction and operation, as well as the advantages of the dielectric type of meter. This instrument finds application in many industries, being used to make rapid, accurate moisture content determinations on a wide variety of products such as: grass seeds, cotton seeds, nuts, and many other granular, powdered, or flaked materials.

TAGIabue Instruments Div., Dept. 67, Weston Electrical Instrument Corp., 614 Frelinghuysen Ave., Newark 5, N. J.

Test Chambers—A data sheet describing the use of Brown instrumentation in the Tenney test chamber is available.

Depending upon the test service for which they are intended, the test chambers described are equipped with the requisite auxiliary equipment and control instrumentation for producing and maintaining the desired trend or status of internal pressure, temperature, or humidity, independently or collectively. Global conditions, from subarctic to tropical, altitudes up to 75,000 ft., and many temperature extremes are said to be readily simulated. Brown instrumentation is used to simulate and maintain required conditions; schematic drawing of on-off system for test chambers; wet-dry bulb control system for test chambers; and program control of temperature and humidity in a test chamber.

Instrumentation Data Sheet No. 11.0-5, Minneapolis-Honeywell Regulator Co., Philadelphia 44, Pa.

Method Sheet for Use of Coleman Nepho-Colorimeter—The method sheet describes the use of the Coleman Nepho-Colorimeter in connection with a count of bacterial growths, determination of sulfur, iron, nickel, manganese, and copper.

Wilkins-Anderson Co., 4525 W. Division St., Chicago 51, Ill.

Method Sheet for Determination of Water (Fischer Reagent Method)—A method of determination of water is fully described with sections devoted to reagents standardization of Fischer reagent, test procedure, and calculation of method to be used. The method employs the WACO Dead Stop Titrator.

Wilkins-Anderson Co., 4525 W. Division St., Chicago 51, Ill.

Instrument Notes

Miniature Compaction Apparatus—Harvard miniature compaction apparatus utilizes a novel method of soil compaction. Consistent moisture-density relationships closely duplicating field compaction curves can be developed in less time and with less effort and material than are possible by present procedures. Unit consists of a tamper, mold, collar remover, and sample ejector.

Soil Testing Services, Inc., 525 N. Noble St., Chicago 22, Ill.

Thermometer for Pressurized Oil Storage Tanks—A new adaptation of the Model 2221 Weston all-metal thermometer accepted by the ASTM as an alternate to mercury-glass angle stem thermometers, has been announced by the manufacturer, Weston Electrical Instrument Corp. Developed to overcome the breakage problem encountered with glass thermometers, these sturdy instruments are designed to fit the sockets already incorporated in many pressurized storage tanks used throughout the petroleum industry. The newly announced Weston thermometer covers a range from 0 to 150 F., with the scale marked in 1 deg. divisions. Accuracy is within 1 deg. over the entire scale.

Weston Electrical Instrument Corp., 617 Frelinghuysen Ave., Newark 5, N. J.

Strain-Gage—A new type of SR-4 bonded resistance wire strain gage, self-compensated for temperature variation, is announced by Baldwin-Lima-Hamilton Corp. The new strain gages are available for use on Dural and Steel in the form of single elements, double elements, and rosettes. Principal advantage of the new selfcompensating gage is elimination of a second strain gage to compensate for temperature changes, thus saving application time and wiring. Basically, the new gage is similar to standard bakelite gages with cupro-nickel wire and its application is by means of the same methods and phenol-resin cement. Gages for two temperature ranges are offered: 50–300 F. and –50 to +300 F. Single-element gages for Dural or Steel are now in stock in four gage lengths: $\frac{1}{4}$, $\frac{1}{2}$, $\frac{3}{4}$, and 1 in. Temporarily, double elements and rosettes will be available on special order.

Baldwin-Lima-Hamilton Corp., Paschall Station P. O., Philadelphia 42, Pa.

Induction Period Calculator—Precision Scientific Co. offers the Induction Period Calculator, a simple device designed to eliminate human error and interpretation in the ASTM test for Oxidation Stability of Gasoline (D 525). It was designed to provide petroleum laboratories with a fast, accurate, reproducible means of calculating the Induction Period of Gasoline. The Calculator consists of two parts: a durable white laminated plastic disk, 15 in. in diameter, and a heavy bakelite template. The template fits over a brass hub which aids in centering the pressure charts. Either the Taylor or Foxboro charts can be used. Calibrations on the plastic disk are printed below the top layer of plastic so that they cannot rub off with wear.

Precision Scientific Co., 3737 W. Cortland St., Chicago 47, Ill.

Flow Table—Precision Scientific Co. has recently produced a mechanical 10-in. diameter flow table for use in ASTM tests of hydraulic cement. Cement testing laboratories of construction companies, state highway departments, and cement manufacturers will find a sturdily built apparatus that conforms precisely to specifications C 230 set by the American Society for Testing Materials. The flow table also meets the requirements of ASTM specifications C 87, C 91, and C 109. In contrast to older hand-operated models the distinctive feature of the new flow table is a $\frac{1}{2}$ -hp. motor geared to operate the drive shaft at 108 rpm. This affects the specified 25 drops in 14 sec. The motor is mounted on a bracket independent of the flow table proper, thus preventing vibration and interference with operation of the test.

Precision Scientific Co., 3737 W. Cortland St., Chicago 47, Ill.

Ductility Testing Machine—A new ductility testing machine providing a total capacity of a quarter of a million pounds pressure and incorporating a unique 5-in. diameter penetrator has been made by

Steel City Testing Machines, Inc., for the Fontana Works of Kaiser Steel Corp. Purpose of the unit is to detect surface and subsurface imperfections in deep-drawing steel over a comparatively large area. The machine is motorized, hydraulically operated, and provides separate controls for clamping the sample and for regulating the penetrating pressure. Penetrating pressures can be applied up to 150,000 lb. while clamping pressures are provided up to 100,000 lb. Equipped with a set of three dies for use with different gages of metal, the machine will test material up to $\frac{1}{4}$ in. in thickness.

Steel City Testing Machines, Inc., 8843 Livernois, Detroit 4, Mich.

INSTRUMENT COMPANY NEWS

*Announcements, changes
in personnel, new plants and
locations, and other notes of interest*

BAUSCH & LOMB OPTICAL CO., 635 St. Paul St., Rochester 2, N. Y. Harry G. Ott, noted optical engineer and assistant to Carl L. Bausch, Vice-President in charge of Research and Engineering at Bausch & Lomb Optical Co., died November 21, 1950. He was 56 years old. He had been a member of the firm's Military Engineering Department for the past eleven years and head of the department since 1948.

HARRY W. DIETERT CO., 9330 Rose-lawn Ave., Detroit 4, Mich., announces the appointment of Frank S. Brewster as general manager. Mr. Brewster was graduated from Michigan State College in 1926. For the past four years he has been sales manager.

FISHER SCIENTIFIC CO., 717 Forbes St., Pittsburgh 19, Pa., announces that J. William Geisler has recently joined the Pittsburgh staff of this firm as petroleum apparatus engineer. Manufacturer of laboratory equipment and chemicals, Fisher has engaged already broad interests by acquiring the highly regarded Tagliabue line of petroleum testing equipment (see Instrument Company News, December 1950, BULLETIN). Mr. Geisler will advise on related matters of engineering, manufacture, and distribution through the five Fisher Plants and through authorized dealers under the registered trade name of "Fisher-Tag."

FISHER SCIENTIFIC CO. opened a new plant in Washington, D. C., December 8. This new scientific supply house will serve as a stocking, shipping, and repairing center for the Atlantic Seaboard area. The plant is located at 7722 Woodbury Drive in suburban Washington. Over an acre of modern shelving is available in the new air-conditioned plant to store the thousands of laboratory apparatus items, instruments, and chemicals. Some

4400 ft. of fluorescent lighting is used in the 50 thousand sq. ft. modern plant of brick, glass, and aluminum. Orders are filled and material distributed from large stocks in Pittsburgh, St. Louis, New York, and Montreal. The Washington plant serves as an advance supply base for the Atlantic Coastal area. Ten trunk lines facilitate phone calls and both teletype and Western Union teleprinter are on hand to speed messages to the company's other stocks and manufacturing facilities in New York, Pittsburgh, St. Louis, and Edgewater, N. J.

Calendar of Society Meetings

"Long" and "short" calendars will appear in alternate BULLETINS. The "short" calendar notes meetings in the few immediate weeks ahead—the "long" calendar for months ahead.

SOCIETY OF AUTOMOTIVE ENGINEERS—Annual Meeting & Engineering Display, January 8-12, Book-Cadillac, Detroit, Mich.

SOCIETY OF PLASTICS ENGINEERS, INC.—7th Annual National Technical Conference, January 18-20, Hotel Statler, New York, N. Y.

COMPRESSED GAS ASSOCIATION—Annual Meeting, January 22-23, Waldorf-Astoria Hotel, New York, N. Y.

NATIONAL CONCRETE MASONRY ASSOCIATION—Annual Meeting and Exhibit, January 22-25, Cleveland Auditorium, Cleveland, Ohio.

AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS—Winter Meeting, January 22-26, Hotel Statler, New York, N. Y.

INSTITUTE OF AERONAUTICAL SCIENCES—Annual Meeting, January 22-26, Astor Hotel, New York, N. Y.

THE ASSOCIATION OF ASPHALT PAVING TECHNOLOGISTS—Annual Meeting, February 4-7, Cosmopolitan Hotel, Denver, Colo.

NATIONAL CRUSHED STONE ASSOCIATION—34th Annual Convention week of February 5, Netherland-Plaza Hotel, Cincinnati, Ohio.

NATIONAL SAND AND GRAVEL ASSOCIATION—35th Annual Convention and NATIONAL READY MIXED CONCRETE ASSOCIATION—21st Annual Convention, February 11-15, Roosevelt Hotel, New Orleans, La.

AMERICAN INSTITUTE OF MINING & METALLURGICAL ENGINEERS—Annual Meeting, February 18-22, Jefferson Hotel, Metals Branch Session, Statler Hotel, St. Louis, Mo.

AMERICAN CONCRETE INSTITUTE—Convention, February 20-22, Hotel St. Francis, San Francisco, Calif.

AMERICAN CONCRETE PIPE ASSOCIATION—43rd Annual Convention, March 1-3, Waldorf-Astoria Hotel, New York, N. Y.

THE PITTSBURGH CONFERENCE ON ANALYTICAL CHEMISTRY AND APPLIED SPECTROSCOPY AND EXPOSITION OF MODERN LABORATORY EQUIPMENT—March 5-7, Pittsburgh, Pa.

American Society for Testing Materials—Spring Meeting & Committee Week, March 5-9, Cincinnati, Ohio.

NATIONAL ASSOCIATION OF CORROSION ENGINEERS—Conference and Exhibition, March 13-16, Statler Hotel, New York, N. Y.

7TH WESTERN METAL CONGRESS & EXPOSITION—March 19-23, Oakland, Calif.

AMERICAN CHEMICAL SOCIETY—119th National Meeting (divided), April 1-5 and April 8-12: Boston, April 1-5; Cleveland, April 8-12.

AMERICAN SOCIETY OF MECHANICAL ENGINEERS—Spring Meeting, April 2-5, Atlanta, Ga.

SCIENTIFIC APPARATUS MAKERS' ASSOCIATION—Annual Meeting, April 15-18, The Greenbrier, White Sulphur Springs, West Va.

AMERICAN MANAGEMENT ASSOCIATION, INC.—20th National Packaging Exposition, April 17-20, Atlantic City Auditorium, Atlantic City, N. J.

THE AMERICAN CERAMIC SOCIETY—53rd Annual Meeting, April 22-26, Palmer House, Chicago, Ill.

AMERICAN FOUNDRYMAN'S SOCIETY—55th Annual Convention, April 23-26, Buffalo, N. Y.

AMERICAN WOOD PRESERVERS' ASSOCIATION—Annual Meeting, April 24-26, Stevens Hotel, Chicago, Ill.

METAL POWDER ASSOCIATION—Annual Meeting, April 25-26, Hotel Cleveland, Cleveland, Ohio.

5TH SOUTH AMERICAN CONGRESS OF CHEMISTRY—May 4-11, Lima, Peru.

FOREST PRODUCTS RESEARCH SOCIETY—Technical Sessions & Wood Products Exhibit, May 7-13, Convention Hall, Philadelphia, Pa.

AMERICAN INSTITUTE OF ARCHITECTS—Convention and National Seminar Meetings, Building Products Exhibit, May 8-11, Edgewater Beach Hotel, Chicago, Ill.

ENGINEERING INSTITUTE OF CANADA—Annual Meeting, May 9-11, Mount Royal Hotel, Montreal, Quebec, Canada.

AMERICAN SOCIETY FOR QUALITY CONTROL—Annual Convention, May 23-24, Hotel Cleveland, Cleveland, Ohio.

SOCIETY OF THE PLASTICS INDUSTRY—Annual National Meeting, May 24-25, Greenbrier Hotel, White Sulphur Springs, West Va.

NATIONAL APPLIED MECHANICS CONGRESS—June 11-16, Chicago, Ill.

American Society for Testing Materials—Annual Meeting, June 18-22, Atlantic City, N. J.

MALLEABLE FOUNDERS' SOCIETY—Annual Meeting June 21-22, The Homestead, Hot Springs, W. Va.

AMERICAN FOUNDRYMAN'S SOCIETY—8th Annual Chapter Officers Conference, June 25-26, La Salle Hotel, Chicago, Ill.

AMERICAN SOCIETY FOR ENGINEERING EDUCATION—Annual Meeting, June 25-29, Michigan State College, East Lansing, Mich.

AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS—Summer General Meeting, June 25-29, Royal York Hotel, Toronto, Ontario, Canada.

National Bureau of Standards Semicentennial

THE year 1951 marks the fiftieth anniversary of the National Bureau of Standards. A large number of the principal scientific and technical organizations of the nation, in recognition of the role of the Bureau in science, have planned meetings in Washington in 1951 to honor the Bureau's Semicentennial, and a calendar of major meetings has been announced. Some of the meetings have regularly been held in Washington in the past; most of them were planned for Washington by the societies in view of the Bureau's Semicentennial.

Corps of Engineers' Method for Design and Control of Asphalt Paving Mixtures*

By Gayle McFadden¹ and Walter C. Ricketts¹

THE Corps of Engineers has conducted an extensive investigational program over the past seven years to obtain data that could be used in formulating design criteria for pavements expected to be subjected to wheel loads far in excess of those for which previous designs had made provision. Among these investigations was one to provide information leading to a simple and rapid method of design and control of asphalt paving mixtures.

The over-all scope of this investigation contained the following general provisions: (1) to select a simple testing device suitable for design and field control of asphalt pavements; (2) to correlate the results obtained by the use of the selected device with field performance for various wheel loads; (3) to establish suitable criteria for asphalt pavements; and (4) to establish the thicknesses of asphalt pavements of known quality required for various wheel loads.

The bases for establishing the general scope of the investigation were: (1) the difference in character and magnitude of wheel loads of airplanes and military vehicles as compared with highway traffic; (2) military airplanes with ever-increasing wheel loads were being planned and constructed; (3) the usual methods employed for design and control of asphalt paving mixtures were not readily adaptable to the preparation and construction of asphalt pavements to meet the requirements in continental United States and in Theaters of Operations for rapid field control; and (4) asphalt pavement of high quality adequate for various wheel loads without overdesign must be provided.

Asphalt paving mixtures were usually designed and constructed following certain criteria and specifications which had proved satisfactory over a long period of time. Each engineer had his own empirical method and employed in some cases various types of testing machines to check design. In most instances there was little or no correlation be-

tween the design and field performance under traffic and there was little or no experience in connection with very heavy wheel loads. The engineer adjusted the paving mixture at the start of construction based on his knowledge of local materials and traffic conditions. The compaction of the pavement was often left to the judgment of the roller operator.

During the recent World War, a different condition developed. It was necessary to expeditiously design and construct asphalt pavements for airfields and cantonment areas to carry loads far in excess of those carried by highways or city streets. Also, the methods of field control usually employed for pavement construction were not considered adequate on jobs which required the use of one and sometimes two asphalt plants producing up to 2500 tons of paving mixtures a day to meet completion dates.

To obtain the necessary data for establishing design procedures, pavement criteria, and adequate field control, investigational projects were initiated by the Office, Chief of Engineers.

INVESTIGATIONAL PROJECTS

The two initial projects consisting of a laboratory study (1943) and field investigation (1944) were authorized and were assigned to the Corps of Engineers Flexible Pavement Laboratory, located at the Waterways Experiment Station, Vicksburg, Miss., for accomplishment. The scope of the over-all investigation was to:

1. Study the existing methods of asphalt pavement design, select a method requiring testing apparatus adaptable to field design and control, conduct a comprehensive laboratory investigation of the testing apparatus, and perform such correlations with other existing apparatus as considered necessary or advisable.

2. Conduct a comprehensive laboratory study utilizing the selected apparatus to compare the test properties of sand asphalt and asphaltic concrete mixtures with aggregate type, gradations, and amount and type of filler as variables. Correlate laboratory compaction of asphalt paving mixtures with densities obtained in the field at the time of construction.

3. Construct a field test section of several types of asphalt pavements in-

cluding surface treatments on three qualities of base and conduct accelerated traffic tests thereon.

4. Conduct final laboratory studies and analyze the data obtained from this investigation and establish satisfactory design criteria and control procedures.

This paper discusses the laboratory work related to the establishment of pavement test properties and the procedures for design and field control of asphaltic concrete paving mixtures. As reference will be made later to pavement criteria established from the data obtained in the over-all investigation, the test section and testing procedures are briefly described. The complete supporting data on which the pavement criteria are based are contained in a published Corps of Engineers report: "Investigation of the Design and Control of Asphalt Paving Mixtures," in three volumes.²

FIELD INVESTIGATION

In 1944 a test track was constructed on a well-drained site at the Waterways Experiment Station. The specific objectives were as follows:

1. To compare asphalt pavement mixtures which have a wide range of physical properties but equal stability values under traffic of 15,000, 37,000, and 60,000-lb. wheel loads (60,000-lb. load on dual tires).

2. To determine the stability values of asphalt pavements satisfactory for these wheel loads.

3. To establish the minimum thickness of asphalt pavements on a high-quality base course, based on a specified stability for the three wheel loads.

4. To determine the optimum asphalt content that will produce satisfactory pavements when submitted to traffic of the three wheel loads listed above.

5. To compare laboratory and field densities.

The field test project (Fig. 1) consisted of two straight parallel tracks each 850 by 60 ft. wide, joined at the ends by turnarounds 175 ft. square. The test tracks were divided longitudinally in three traffic lanes—18, 20, and 22 ft. wide, respectively, for the 15,000,

NOTE.—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communications to ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa.

* Extended abstract of paper presented at the Session on Bituminous Paving Mixtures held at the First Pacific Area National meeting of the Society, San Francisco, Calif., October 10-14, 1949.

¹ Office, Chief of Engineers, Department of the Army, Washington, D. C.

² Copies of the report, at the cost of reproduction, may be obtained from the Director, Waterways Experiment Station, Vicksburg, Miss.

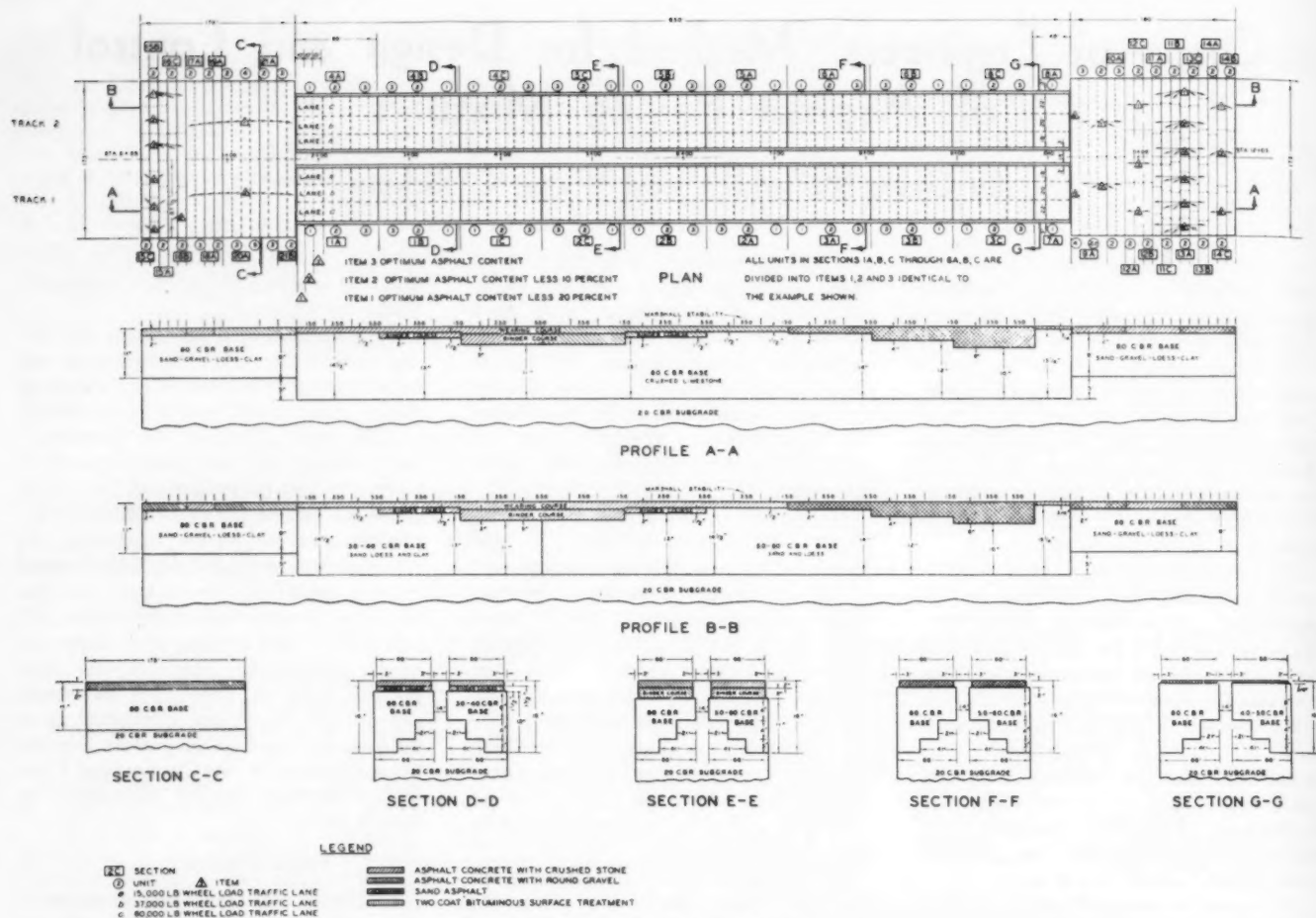


Fig. 1.—Plan and Profiles of Test Section.

37,000, and 60,000-lb. wheel loads. The total thickness of the pavement and base for each lane was designed in accordance with the design criteria shown in the Corps of Engineers Engineering Manual (1944), plus about 20 per cent. The base course for the asphaltic concrete sections was a good grade of crushed stone with a minimum California Bearing Ratio (CBR) of 80. The pavement structure was purposely overdesigned to eliminate possible base failures which would distort subsequent study of the effects of traffic on the asphalt pavement.

The test track and turnarounds were divided into sections which were progressively subdivided into subsections, units, and items.

This summary deals only with the asphaltic concrete subsections 1A, 1B, 1C, 2A, 2B, and 2C of Track No. 1. each 90 ft. in length. Subsections 1A and 2A were paved with a single course of asphaltic concrete $1\frac{1}{2}$ in. thick, subsections 1B and 2B, in two courses totaling 3 in., of which $1\frac{1}{2}$ in. was binder course and $1\frac{1}{2}$ in. was surface course, and subsections 1C and 2C were paved in two courses totaling 5 in. of which 3 in. was binder course and 2 in. was surface course. Each subsection was divided

into units 30 ft. in length. Units Nos. 1, 2, and 3 were designed for 150, 350, and 550-lb. Marshall stability values, respectively. Each unit was divided into three items 10 ft. in length in order to determine the relationship between the optimum asphalt content as determined by the Marshall stability test and the optimum required by traffic

compaction for the three listed wheel loads. The theoretical optimum asphalt content was determined by the Marshall machine and, to provide a test range, the asphalt content of items 1, 2, and 3 were established at 20 per cent below optimum, 10 per cent below optimum, and optimum, respectively.

The Marshall machine was used to

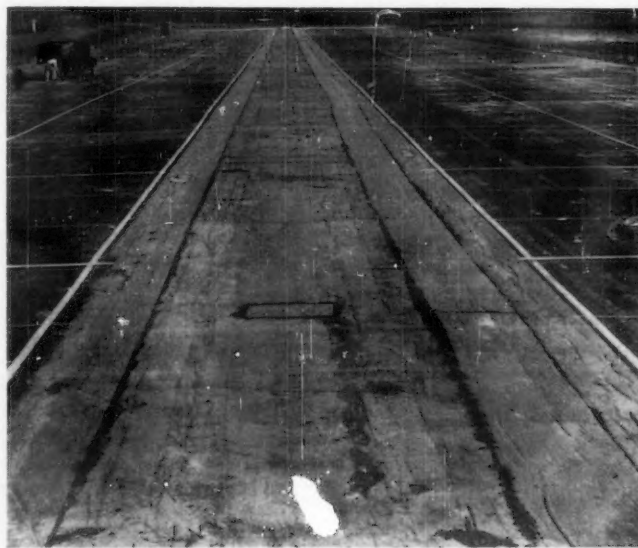


Fig. 2.—View of Completed Test Section.



Fig. 3.—View of Model Super C Tournapull Connected to Specially Constructed Load Cart Used for 37,000-lb. Single and 60,000-lb. Dual Wheel Load Traffic Tests.

determine stability and optimum asphalt content of both binder and surface courses. The aggregate used in producing the surface course mixtures was well graded conforming with Corps of Engineers' 1944 specifications for $\frac{3}{4}$ in. maximum with about 50 per cent of the material being retained on the No. 10 sieve. The gradations in general follow Fuller's curve.

The amount of commercial limestone filler passing the No. 200 sieve used in these paving mixtures was in the order of 3.5 per cent for 150-lb. stability mixtures, 6 per cent for 350-lb. stability mixtures, and 8 per cent for 550-lb. stability mixtures.

Subsections 1A, 1B, and 1C and subsections 2A, 2B, and 2C were paved with mixtures containing commercially produced aggregates consisting of crushed limestone and sand and uncrushed gravel and sand, respectively.

The asphalt used was a high quality and had a penetration of 120–150.

The same type aggregates, asphalt, and mineral filler were used for producing the binder course mixtures. The asphalt content was in the order of about 4.5 per cent instead of the range of 4.5 to 7.5 per cent used in the surface course. The aggregate portion was well graded from $\frac{3}{4}$ in. down with about 60 per cent retained on the No. 10 sieve. The percentage of material passing the No. 200 sieve was also reduced slightly as compared with the surface courses.

The paving mixtures were produced by a small standard batch plant and the pavements were constructed in accordance with standard paving equipment and practices. Figure 2 shows the completed test track.

TRAFFIC TESTS

Traffic testing was initiated on the 15,000-lb. lane in May, 1945, and 3500 coverages were applied as of October, 1945. A coverage is defined as one load application over every point in a given area. A 12-cu.-yd. scraper was used for the 15,000-lb. wheel load tests. The scraper unit was loaded in such a

Most of the testing was conducted at temperatures from 100 to 125 F.

The experience obtained from the traffic testing of the 15,000-lb. lane indicated that 1500 coverages were sufficient for the purpose of the investigation. The physical properties did not change in the unfailed pavements by the application of additional coverages, and, as in the case of all accelerated tests, it is impossible to evaluate the effect of weather and time.

The tracking pattern was so arranged

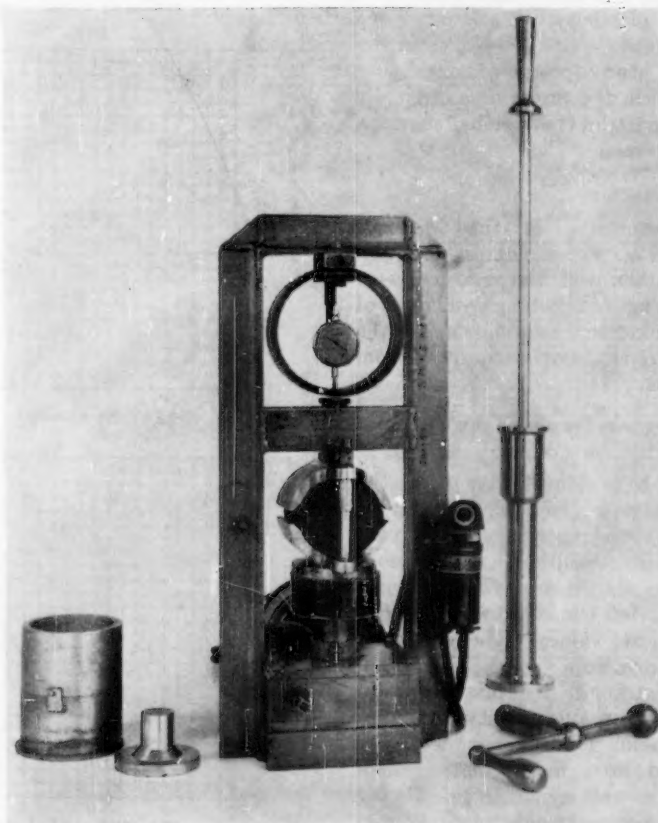


Fig. 4.—Marshall Stability Apparatus for Field or Laboratory Use.

manner that each wheel carried 15,000 lb.

A specially designed runway load testing device (Fig. 3), in which the actual landing gear assembly for the test load could be placed, was used for testing the 37,000 and 60,000-lb. lanes.

Tests with the 37,000-lb. single wheel load were started in September, 1945. Fifteen hundred coverages were applied to the 37,000-lb. lane in 1945 and summer of 1946.

Tests using 56-in. dual wheels (B-29 airplane) installed in the testing device and loaded to 60,000 lb. were started in August, 1945. Fifteen hundred coverages were applied to the 60,000-lb lane in 1945 and summer of 1946.

All traffic testing was conducted when the pavement temperature was 90 F. or higher with a maximum of 140 F.

as to avoid the effect of repeated coverage of one wheel on the same area. The direction of the traffic was reversed at regular intervals.

Pavement cores were taken at frequent intervals during the testing for laboratory examination, and an accurate record supplemented with photographs was kept of all unusual conditions that developed in the pavements.

LABORATORY STUDY

The first objective under the laboratory study was to select or develop, if necessary, a simple and highly portable testing machine.

The Marshall testing machine, having apparently met the desired requirements, was tentatively selected. The final selection was to be based on the ability of the machine to satisfactorily

measure properties of a paving mixture when compared with other suitable types of testing machines.

Comparative laboratory tests were performed on a range of asphalt paving mixtures using both the Hubbard-Field and Marshall machines. This work indicated that the Marshall machine was satisfactory. The values obtained could be used for selecting proper asphalt content and reflected variations in gradation of aggregate, character of aggregate, variations in filler content, penetration of asphalt, and also measured the plasticity of a compressed paving mixture.

As the study progressed, revisions were made in the procedures originally used, and parts of the Marshall machine were redesigned.

Marshall Machine:

The description of the Marshall machine (Fig. 4) and auxiliary equipment together with the methods used for preparing and testing specimens of paving mixtures, are shown in the published report² but are omitted from this summary.

DISCUSSION OF PRELIMINARY LABORATORY WORK

In order to evaluate and compare the quality of two or more asphalt paving mixtures it was necessary to develop yardsticks for measuring pertinent properties of compressed paving mixtures and to express the selected properties in numerical values. The Marshall machine provided a yardstick for measuring the strength and plasticity. In addition, the specific gravity of a compressed paving mixture can be determined and used in calculating such properties as unit weight in pounds per cubic foot, per cent voids total mix, and the percentage of total voids filled with asphalt.

Other factors which are known to affect the quality of an asphalt paving mixture were also studied, such as asphalt content, gradation and type of aggregate, quantity and type of filler, and penetration of asphalt cement, but discussions are omitted from this presentation (see Figs. 5, 6, and 7).

CONCLUSIONS ON LABORATORY STUDY

The conclusions listed below are believed to be justified as the result of the laboratory tests:

1. The Marshall machine measures in a satisfactory manner both internal friction of the aggregate fraction of the mixture and the adhesion of the asphalt.
2. The Marshall stability values are indicative of the relative strength of basically different asphaltic concrete pavements.

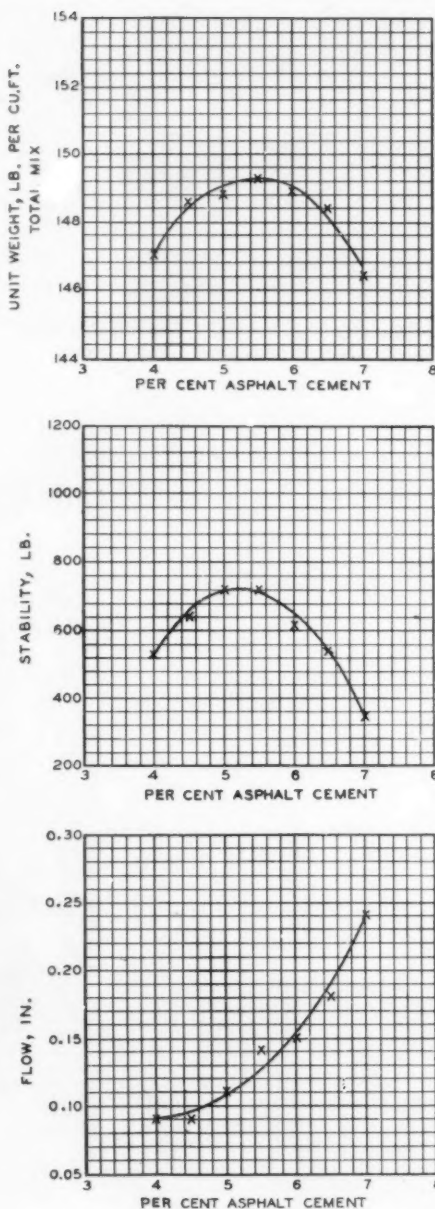


Fig. 5.—Typical Curves Test Properties versus Asphalt Content (Asphaltic Concrete).

3. The flow value is a measure of the plasticity of an asphalt pavement. Mixtures of equal stability but containing varying amounts of asphalt may be compared on the basis of flow values.

4. Increases of asphalt in increments to a given mixture will produce an increase in the total weight to some maximum value after which increases of asphalt will cause the total weight of the mixture to decrease.

5. In general, asphaltic concrete pavement with relatively low void content gave higher Marshall stability values.

6. The stability values of an asphaltic pavement were increased by addition of coarse aggregate to a maximum of about 65 per cent of the total aggregate.

7. The stability values of an asphalt pavement were not materially changed by the type of aggregate when present

in the mixture in amounts less than about 40 per cent.

8. In general, asphaltic concrete mixtures containing crushed coarse aggregate produced higher stability values than uncrushed aggregate.

9. In general, the addition of higher quality filler (material passing No. 200 sieve) to an asphaltic concrete mixture increases the stability, reduces the voids in the aggregate portion of the mixture, and tends to reduce the amount of asphalt required for maximum stability. The amount of filler to be added to an asphaltic concrete producing the most suitable pavement has not been determined by this laboratory study.

10. The stability values obtained by the use of the Marshall machine reflect changes in asphalt content, quantity and type of filler type and gradation of aggregate, and changes in penetration of asphalt.

TEST PROPERTIES OF ASPHALTIC CONCRETE SURFACE COURSES

From all data obtained the following criteria have been established for asphaltic concrete surface course pavements for wheel loads between 15,000 and 37,000 lb. on single and 60,000 lb. on dual wheel loads with gross tire pressure between 55 and 100 psi. net pressures as high as 140 psi.

Stability minimum.....	500 lb.
Flow maximum.....	20
Per cent voids, total mix.....	3 to 5
Per cent voids, filled with asphalt.....	75 to 85

The pavement thicknesses conforming to the above criteria and considered suitable based on this investigation for the wheel loads used in the traffic testing, when placed on base courses of 80 CBR values or better, are as follows:

Wheel Loads, lb.	Total Pavement Thickness, in.	Binder Course Thickness, in.	Surface Course Thickness, in.
15 000...	2	1½	2
37 000...	3	1½	1½
60 000 (dual wheels)	3	1½	1½

All binder courses in the asphaltic concrete section performed in a satisfactory manner.

The importance of determining the proper amount of asphalt to use in a pavement has long been recognized. The test properties (Fig. 5) used in connection with the Marshall machine for selecting the optimum asphalt content are unit weight in lb. per cu. ft., per cent voids, stability, per cent voids filled with asphalt, and flow.

The method used for selecting the optimum asphalt content for a given mixture is based on the Marshall stability values obtained on laboratory specimens whose compositions are the same except the asphalt contents are varied in 0.5 increments. The other values are calculated from the other laboratory determined data. The values for each property are plotted against asphalt content. In this typical example the per cent asphalt at the peak of each curve is as follows:

Stability.....	5.3 per cent
Unit weight.....	5.3 per cent
Voids.....	5.8 per cent
Voids filled with asphalt...	6.3 per cent
Average.....	5.7 per cent

The value for flow is not used in selecting asphalt content.

The individual test properties at the average asphalt content of 5.7 per cent are then re-examined to determine how closely they agree with the established criteria. In this example and at this asphalt content the flow value is 14, stability is 700 lb., voids total mix is 4.1 per cent, and voids filled with asphalt

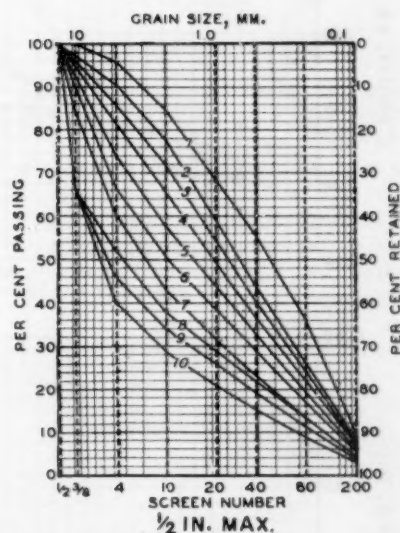
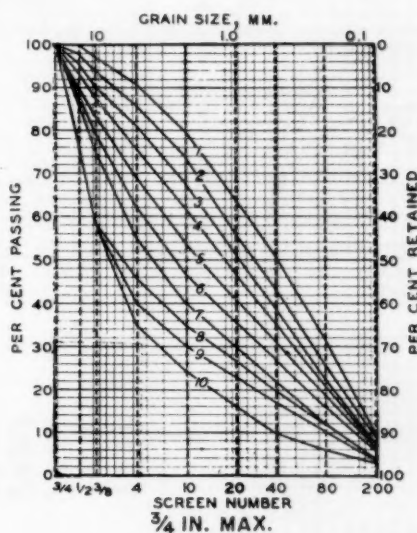
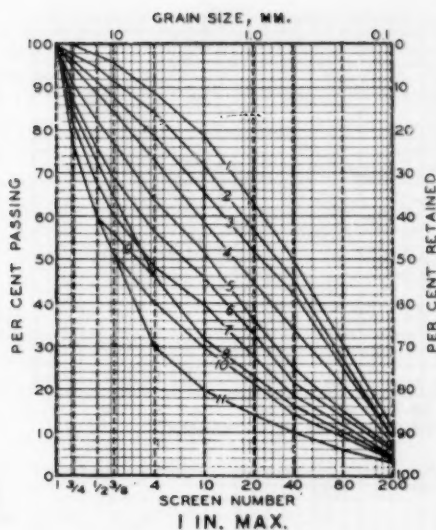


Fig. 6.—Aggregate Grading Charts (Asphaltic Concrete).

Note.—Screen numbers refer to U. S. Standard sieve.

76 per cent. All are in agreement with the criteria. In event the test properties of the mixture do not meet the criteria, adjustments in gradation or proportions, or selection of other materials would be indicated.

FIELD CONTROL OF PLANT MIXTURES

The use of the Marshall machine in the field for plant control of paving mixtures does not preclude the need for the usual methods employed such as making screen analysis of bin samples and aggregates received, extraction tests of finished mixtures, and other plant controls. However, it is possible to make and test specimens of hot paving mixtures at the plant in about 30 min. as frequently as necessary. In event the pavements do not conform to the established criteria, corrective measures may be taken before considerable tonnage is produced and laid.

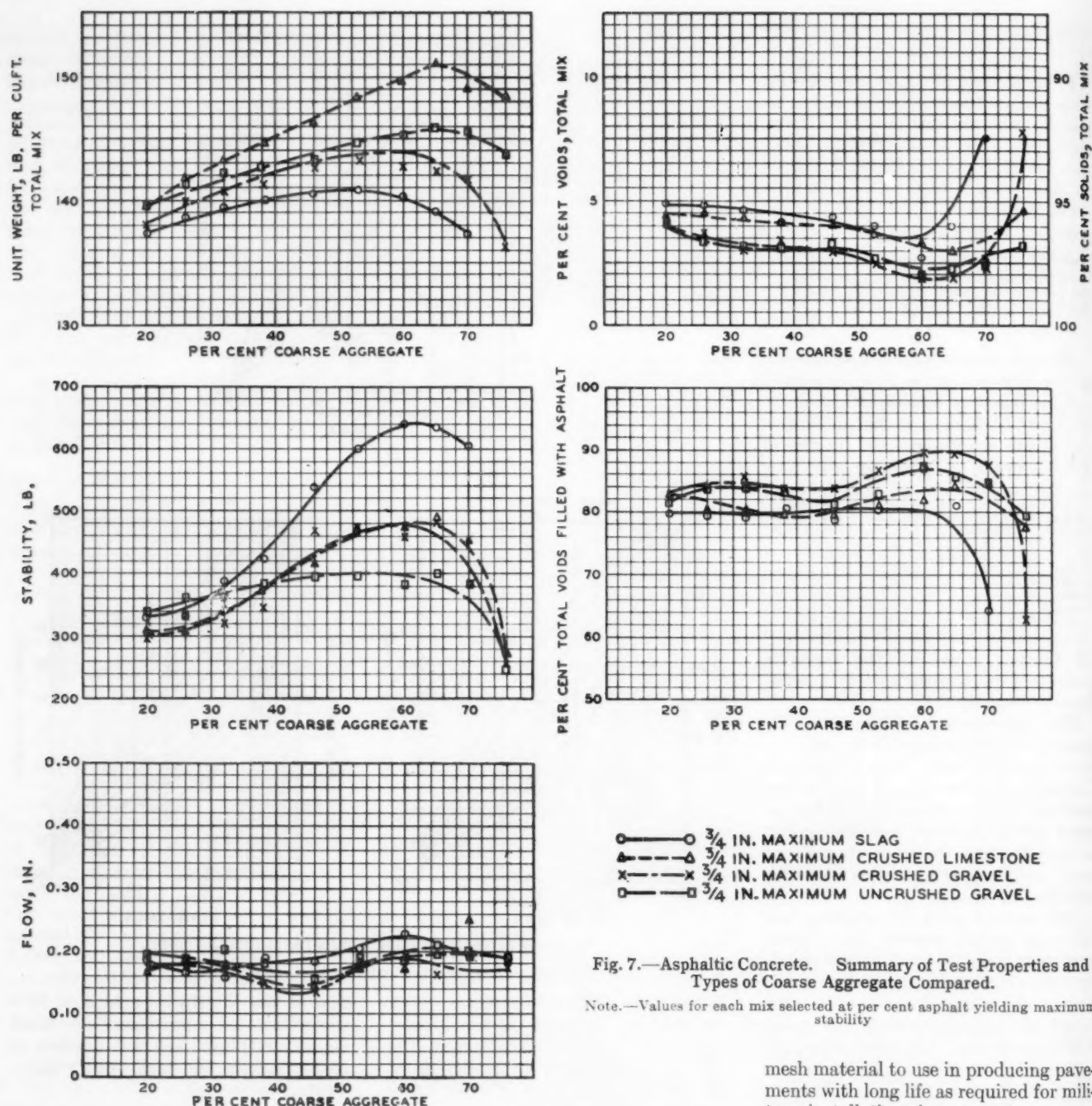
It is believed that the proper applica-

tion of the Marshall machine to field control is a vast improvement over usual methods employed and will do much to insure uniform and high quality pavements.

CONTROL OF PAVEMENT CONSTRUCTION

All other conditions being equal, the results of the investigational work indicate that for a given mixture stability increased with density except for mixtures containing excessive asphalt contents.

In order to control the amount of rolling necessary to secure a finished pavement conforming with the established criteria, it was necessary to determine by field tests the percentage of pavement density required as compared with the density of a specimen prepared for test by the standard procedures. By a series of field tests, it was established that pavements could be readily rolled by the use of standard equipment



and practices to 98 per cent of the density obtained by the compactive effort used to prepare a test specimen by the standard procedure.

The amount of rolling may be readily field controlled by determining the density of samples of pavement either cored or cut from pavement during construction and will insure finished pavements conforming with the established criteria.

ADDITIONAL INVESTIGATIONAL WORK

The present Marshall machine and procedures are applicable for conducting tests on paving mixtures containing aggregates of 1-in. maximum size or less. Laboratory and field work are in

progress to develop a mold and procedures suitable for use with the present Marshall machine and capable of testing mixtures containing aggregates up to 2½ in. maximum.

The larger mold will be used primarily to establish test properties and criteria suitable for asphaltic binder and base courses for heavy wheel loads.

There are many opinions as to the exact percentage of material passing the No. 200 sieve that should be used to produce the most satisfactory pavements. Experience indicates that pavements containing excessive amounts of No. 200 mesh material tend to crack with age. The need to establish the most suitable percentages of No. 200

mesh material to use in producing pavements with long life as required for military installations is apparent.

The percentage of No. 200 mesh material in a runway pavement is particularly critical as the middle third of a runway receives most of the traffic. This leaves considerable areas which do not receive the beneficial kneading of concentrated traffic and may crack if not properly designed.

Laboratory work is now in progress to develop a type of machine that will indicate the permissible degree of flexibility of an asphalt pavement. It is hoped that with data obtained by the use of a flexibility machine the most satisfactory limits for material passing No. 200 sieve to be used in preparing asphalt mixtures may be definitely established.

Since the start of this investigation airplanes weighing, when loaded, over 300,000 lb. have been constructed, and tires capable of being inflated up to 300 lb. are distinct possibilities as standard equipment for future airplanes. Additional investigational work is now under way to determine whether modifications of the established pavement criteria are required for pavements subjected to heavier loads (excess of 60,000 lb. on dual wheels) and high-pressure tires.

SUMMARY

The Marshall machine, being highly portable and requiring a minimum of ordinary type auxiliary laboratory equipment, is equally adapted for use

in a central or a field laboratory for either design or plant control.

Specimens may be easily prepared and tested in a minimum of time.

The pavement criteria established are in terms of simple numerical values which may be readily determined by the use of the Marshall machine and the established design procedures.

By the proper use of the Marshall machine and procedures, a high-quality pavement adequate for the demands of traffic may be designed and constructed eliminating the necessity for "guesses" to be made by the engineer in charge.

The test procedures permit local aggregates to be evaluated as compared with more expensive shipped-in aggregates.

It should be pointed out that the

method for design and control of asphalt paving mixtures employed by the Corps of Engineers is empirical and in it the Marshall machine and procedures are used as a yardstick for evaluating the paving mixture.

The pavement criteria were established as the result of traffic tests on pavements designed and evaluated by the Marshall machine and procedures using wheel loads up to and including 60,000 lb. on dual wheels.

It is considered that before any method either empirical or theoretical can be used with confidence for designing economical asphalt pavements adequate for very heavy loads, correlation of the effect of actual traffic on pavements designed by the method is necessary.

DISCUSSION

MR. L. E. GREGG.¹—The comments that I have to make are related to some of the work done by the Kentucky Department of Highways Research Laboratory over the past several years, in which an attempt is being made to correlate the Marshall Stability Test with other test procedures and with field performance.

The procedure consisted of about the same techniques that were used by the Corps of Engineers, but on a smaller scale. Samples were prepared on construction projects, and others were prepared in the laboratory using comparable mixes. Some of these were exposed outdoors for various periods of time at the laboratory. Some were tested immediately. Other specimens were cores cut from the pavements immediately after they were constructed, and still more cores were taken at various intervals after the pavements were in service.

Now, the value of this method depends upon the accuracy with which the prepared samples represent the conditions of the pavement as it exists after construction. Considerable effort was directed toward the evaluation of samples from this standpoint, as well as from the standpoint of test results and pavement correlation.

We found, in our tests, somewhat the same thing as the Corps of Engineers found—that the densities obtained by the Marshall machine were not equal to those obtained with the usual construction equipment. Therefore we are revising the compaction procedure to some extent, but not to the extent that

it was done in the cases described by the authors.

The compaction procedure as we use it still consists of impact blows without distributing each blow over the entire surface of the sample. In other words, the original 2-in. face of the compaction hammer was retained, and this was supplemented by a static load following the impact loads. However, the static load was increased to 7000 lb. whereas originally it had been 5000 lb.

Even with this increase in compactive effort, we found that the average density of mixes prepared in this way was about 5 per cent lower than that usually obtained on the road. We have not yet worked out a procedure for increasing this to the right value, but we do know that by using the Corps of Engineers' procedure we usually get densities in excess of those obtained on the road.

We were concerned somewhat about the crushing of aggregates under the impact blows, and we found through experimental work in the field and in the laboratory that actually the amount of crushing in the finished sample was not equal to the crushing of the aggregate under the rollers. However, the only way we could achieve this condition in the laboratory was by wasting the upper material on samples as the faces were leveled off, following compaction under the hammer and prior to static compaction.

For these comparisons, samples were taken from pavements laid over various types of bases—water-bound macadam, bituminous, portland cement concrete, and others—and almost invariably the crushing under the roller exceeded that on the comparable sample prepared by the laboratory compaction method.

We have experienced considerable difficulty in determining the significance of stability numbers, particularly for some types of mixes. One outstanding example was a pavement with 85 to 100 penetration asphalt cement in combination with uncrushed gravel. Such an aggregate is outside our specifications, but it was tried on an experimental basis for the reason that in some sections of the state we have an abundance of this coarse material that can be obtained at reduced cost provided it doesn't have to be crushed.

For the samples prepared by laboratory compaction as well as those cut from the pavement immediately following construction, the stability numbers were considerably lower than 100, and most of them were below 50. Still, the pavement performed satisfactorily on a relatively heavily traveled road with wheel loads running as high as 10,000 to 12,000 lb.—the average being lower since that is somewhat in excess of our legal limit. There is no evidence of rutting or shoving. In tests on the cores that have been taken since, the stability numbers have increased greatly, but that was not unusual. It was unusual in the percentage of increase, but increases in stability numbers over a period of time are the usual thing rather than the exception.

There are other ways in which some of our conditions have not conformed with the Corps of Engineers' criterion, and yet we have had satisfactory service from the pavements. For example, there have been a few cases where our flow values have exceeded the 0.2-in. limit set by the criterion, and the percentage voids in the compacted mix often exceeds five—sometimes going as high

¹ Associate Director of Research, Kentucky Department of Highways, Highway Materials Research Lab., Lexington, Ky.

as 12 and 13 per cent—without any detrimental effect on the pavement that we have been able to observe.

We do feel that the method is worth while. It is empirical, as the authors have pointed out. It does require correlation with field conditions. That is the principal thing which we lack. Yet we feel that the test does have possibilities, and establishing significance for it may be only a matter of making a sufficient number of tests and accumulating a vast amount of data as done by the Corps of Engineers.

MR. C. E. PROUDLEY² (*presented in written form*).—The data and conclusions presented in the paper by Messrs. McFadden and Ricketts are so comprehensive that it will not be possible here to do more than select a few points to discuss in which I have special interest.

We agree, as a result of our own experience, with the conclusion that the Marshall equipment compares favorably with the Hubbard-Field equipment as a measure of stability, also, that either method will reflect changes in type and gradation of aggregate, asphalt content, penetration of asphalt, and the quantity of No. 200 mesh material. The Marshall apparatus has the additional advantage of portability and a means for measuring a property referred to as plasticity.

It is our observation, however, that the Hubbard-Field method is somewhat more sensitive both in the 2-in. diameter specimen and the 6-in. modification. Values obtained by the North Carolina laboratory in Hubbard-Field tests range from less than 100 lb. for sand-asphalt in eastern North Carolina to over 9000 lb. for bituminous concrete in western North Carolina. Although such sensitivity may not be necessary for field control it is helpful in laboratory design work whenever the stability curve has a tendency to be inconveniently flat. It was observed that the Marshall method in our tests gave from 15 to 40 per cent of the value in pounds of the values obtained on the same mixtures by the Hubbard-Field method depending primarily upon the shape or angularity of the aggregate particles. Our comparative tests are not sufficiently extensive to warrant the establishment of definite comparisons.

Designing bituminous paving mixtures by the use of stability data is relatively new in North Carolina and we have not progressed to the stage of controlling plant performance by continuous stability testing, therefore, we do not need portability and are developing most of our information by means of the Hubbard-Field apparatus.

² Chief Materials and Test Engineer, North Carolina State Highway and Public Works Commission, Raleigh, N. C.

It is our procedure at present, which is in the process of development, to secure representative samples of the materials which it is planned to use on a job and have the laboratory make a series of tests to ascertain whether sufficient stability can be developed to satisfy what we estimate will be the requirement of the anticipated traffic, and also, to secure the best combination of aggregates and percentage of bitumen which will yield the most satisfactory properties.

No doubt everyone will agree that sampling will still require the utmost care and attention in order to avoid nonrepresentative test results. As pointed out by the authors of the paper, the optimum amount of bitumen is indicated by the stability, unit weight, voids in aggregate, and percentage of voids filled with bitumen. Although chance variations in the bitumen content and gradation of the aggregate under job conditions in the field may not affect the stability of the resultant pavement to an important degree, even though the preliminary laboratory tests to establish the job formula might so indicate, nevertheless there are precautions which will have to be prescribed, eventually, in connection with the stability method of job control. We have found that it was not possible to secure the same stability value from reheated mixtures and that the apparent stability increased with lapse of time, no doubt due to hardening of the asphalt by oxidation, absorption, or other slow changes in characteristics of the component materials. Since plant-tested samples will give lower average stabilities than any later test, the control is on the safe side. Details with regard to the securing and handling of samples for job stability control should be kept in mind by members of Subcommittee B-15 of Committee D-4 as an item to be included in ASTM Tentative Methods of Sampling Bituminous Paving Mixtures (D 979-49 T).³

Although excellent work has been reported on the load-carrying capacity of bituminous mixtures of various stabilities and thicknesses, it will still remain for many State Highway Departments when establishing criteria for their own design work, to make studies under the climatic conditions and for traffic and aggregates which they will encounter locally. Since North Carolina, like most other states, cannot afford to conduct an elaborate investigation such as that reported by the authors, we plan to accumulate data as we go along. Whenever new bituminous pavement is constructed we determine the characteristics of the paving mixture either

³ 1949 Book of ASTM Standards, Part 3, p. 1088.

before or after the job is built. Traffic counts are being recorded continuously on all primary and many secondary highways therefore it will be necessary only that we measure the service performance in some way that can be expressed numerically. We have in process of construction a one-wheel roughometer of the Bureau of Public Roads design. Annual, or more frequent, readings on the bituminous pavement, starting immediately after it is opened to traffic will, we hope, furnish us with sufficient data for an enlightening study of the design of pavement and design of paving mixtures on service performance.

MR. B. JAMEYSON⁴ (*presented in written form*).—The data accumulated by the Army Engineers during this investigation probably represent the most extensive study of asphalt pavements yet undertaken. Considerable practical information has been and will be obtained from this study. As stated by the paper the first objective under the laboratory investigation was to select or develop a simple and easily portable testing machine. To this end the Marshall test was chosen. One of the reasons stated for using this test was that it compared favorably with the results of the Hubbard-Field test and that the Hubbard-Field machine was typical of the type that satisfactorily measured the pertinent properties of an asphalt paving mixture. It is agreed that the Marshall test results and those of the Hubbard-Field test are probably comparable. However, there is a large and increasing group of engineers who feel that the Hubbard-Field test does not measure the pertinent properties of an asphalt pavement. I believe that neither of these tests gives due weight to the frictional resistance properties of bituminous mixtures. In other words the data obtained are no more than could be obtained from an unconfined compression test which measures predominantly tensile strength (cohesion).

We have all learned that any test method which has been widely used and with which we are personally experienced is a valuable tool for guiding our thinking when new and untried situations develop. On the other hand, we are inclined to allow our experience and understanding to influence the meaning of numerical test results obtained from empirical tests. Thus, a numerical test result does not always mean the same nor is it handled the same by the neophyte as the experienced engineer. The Marshall test appears to suffer from this shortcoming, as some engineers now becoming acquainted with the test are having difficulty in interpreting the

⁴ California Research Corp., Richmond, Calif.

test results in light of their field experience.

The Marshall test measuring predominantly cohesion will, in many cases, indicate an "optimum asphalt" content which if used will produce an unsatisfactory asphaltic mix. This is especially true in dense, uncrushed, high surface area type mixes. Mixes utilizing this "optimum asphalt" are sometimes very rich and failure occurs due to rutting or shoving. Other tests measuring both cohesion and internal friction of a given mix would have shown that the maximum cohesion occurred at an asphalt content which produced a dangerously low value of internal friction. Therefore, a slightly lower asphalt content, while not producing as high a value of cohesion, would have had a considerably higher value of internal friction and would have been satisfactory. Our field experience indicates that both factors (cohesion and internal friction) are necessary to properly design asphaltic mixes in the laboratory.

One other shortcoming of the Marshall test is the need for testing eight specimens to determine Marshall stability. This fact in itself indicates that a more reproducible and precise test is desirable. Therefore, while the apparatus does possess the advantage of simplicity and portability desired for use during military operations, it is believed that above-mentioned shortcomings outweigh this fact.

MESSRS. GAYLE MCFADDEN AND W. C. RICKETTS (authors' closure). — The thoughtful discussions of Messrs. Gregg, Proudley, and Jameyson are considered valuable and are appreciated.

The investigation that led to the development of the Marshall machine was directed toward establishing pavement criteria and test procedures for constructing pavements suitable for traffic of very heavy military planes and military vehicles. As pavements used in permanent military installations must possess long life and low maintenance costs, the study was limited to hot plant mix pavements using penetration grades of asphalt and well-graded aggregates.

It is believed that some adjustment in the Marshall test values and procedures will be necessary for design of pavements in which asphaltic binders other than penetration grades and poor quality aggregates are used and when there is a wide variance in wheel loads and traffic conditions from those used in the overall investigation. (See footnote 2 of the paper.)

The changes made in the Marshall test procedures by Mr. Gregg, so the results would be representative of traffic conditions and local aggregates

in Kentucky, are considered sound and indicate the utility and adaptability of the Marshall machine.

It is to be noted that Mr. Jameyson's discussion contains the following criticisms of the Marshall method:

1. The stability value determined by the Marshall machine does not give due weight to the frictional resistance properties of bituminous mixtures.

2. The numerical Marshall test results do not always mean the same nor are they handled the same by the neophyte as by the experienced engineer, and some engineers are having difficulty in interpreting the results in light of their field experiences.

3. The Marshall test measuring predominantly cohesion will, in many cases, indicate an optimum asphalt content which if used will produce an unsatisfactory asphaltic mix, particularly in dense, uncrushed high surface area type mixes.

4. One other shortcoming of the Marshall test is the need for testing eight specimens to determine Marshall stability.

Each of Mr. Jameyson's criticisms are discussed in the paragraphs that follow in the same order as listed above.

1. Figure 7 shows the test properties of specimens in which the percentages of $\frac{3}{4}$ -in. maximum-sized aggregate retained on the No. 10 sieve was varied in 5 per cent increments from 20 per cent to 70 per cent. The asphalt content used in each mix was the amount which produced maximum stability, and various types of aggregates were used, namely, slag, crushed limestone, crushed gravel, and uncrushed gravel.

A digest of the stability curves shown on Fig. 7 will disclose that the Marshall test measures frictional resistance as:

(a) The stability values begin to increase when the mixtures contain about 35 per cent of coarse aggregate (material retained on No. 10 sieve). (It is well known to experienced designers of bituminous pavements that the coarse aggregate content of a pavement must exceed about 35 per cent before interlocking of coarse aggregate takes place.)

(b) The stability values of the specimens containing uncrushed gravel did not increase materially due to the lack of angularity of the aggregate while slag specimens had the highest stability values due to the irregular and angular characteristics of the material.

(c) The specimens containing crushed gravel and crushed limestone increase in stability in proportion to the percentage of coarse aggregate from

about 35 per cent up to about 65 per cent.

2. It is the opinion of the authors that the design of asphalt pavements should not be left to the "neophyte." In connection with the interpretation of Marshall test results, it has been pointed out that the pavement criteria were established for construction of facilities suitable for heavy military planes and heavy military vehicles and may require adjustment when applied to design of pavement for other types of traffic and job conditions.

3. It is assumed that Mr. Jameyson is referring to dense, uncrushed, low-surface area mixes, as crushed aggregates normally have higher surface areas than uncrushed aggregates.

It is well known that rich mixtures are those in which the voids are overfilled with asphalt. The established criteria include definite requirements for voids and percentage of voids filled with asphalt. Although not mentioned in the paper, an adjustment in the gradation of the aggregate is necessary when a given mixture does not conform to the voids and percentage of voids filled with asphalt requirements.

A digest of Fig. 5 will disclose that the stability of a rich hot plant mixture (over optimum asphalt) would never be at the peak of the stability curve or conform to the requirements for voids and percentage of voids filled with asphalt and hence would not be considered satisfactory.

Mr. Jameyson may be referring to types of pavement using cut-back asphalts or types of binder other than penetration grades. The applicability of established pavement criteria to such types is not known, as only penetration grades were used in the investigational work.

4. Engineers familiar with laboratory work recognize that the personal equation always enters into the preparation of test specimens. As the laboratory work conducted in connection with over-all investigation was directed toward the selection of testing equipment and establishing pavement design criteria, eight specimens were made to minimize the personal equation.

It is the opinion of the authors that normally eight specimens would not be required for each stability test in designing an asphalt pavement. However, the same procedure as used in any other type laboratory work, namely, rejecting test results which are widely divergent from the average values of all specimens and preparing new specimens for those in which defects are discovered should be followed in preparing specimens for the Marshall test.

Results on Testing Surface Waterproofings*

By F. O. Anderegg¹

SYNOPSIS

Methods of testing surface waterproofers reported at the 1948 Annual Meeting of the Society² represent a phase of the investigations into masonry structures carried out over the past twenty-five years. These methods have now been employed in evaluating a much larger number of cement type and solvent system colorless waterproofers and have been extended to several emulsion waterproofers, to silicone resins, and to sprayed-on stuccos. Additional field studies have shown a good correlation with the laboratory results. Several of those previously reported continue to give excellent performance, while some members of the new types tested seem to show good promise. The greatest hazards seem to lie in the pressures developed by efflorescent salts, by rain erosion, and by oxidation.

It will be remembered that the author proposed the following definitions in a previous paper on "waterproofing" materials² by which, when the material is applied under

- "2. Egress of visible moisture under a pressure, say, of 4 ft. of water from behind (at any time after the first month)."

A considerable number of additional

viously described, the effect of the waterproofing treatment on transpiration, on resistance to efflorescent pressure, and on weathering in the presence of soluble salts has been determined for two or more examples of each of five types of waterproofers.

The use of single bricks for making these tests, rather than going to brick assemblages, has been adhered to for the following reasons:

1. The far greater ease of making the tests with single bricks appreciably reduces costs.
2. With proper care, bricks can be selected with pore openings which can readily be observed and measured, whereas the size of the openings between bricks and mortar in assemblages is

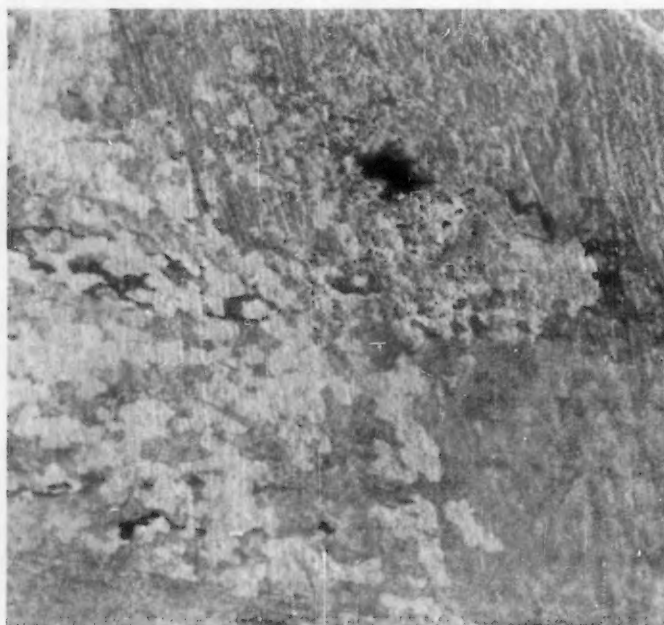


Fig. 1.—Exposure Through Two Winters Has Resulted in Scaling.



Fig. 2.—Erosion of a Material Containing Too Little Cement.

proper conditions to suitable types of masonry, will prevent:

- "1. Ingress of more than 1 lb. of water per square foot in the first hour under a head of $\frac{1}{4}$ in. water, and after one month or more of aging.

NOTE.—DISCUSSION OF THIS PAPER IS INVITED, either for publication or for the attention of the author. Address all communications to A.S.T.M. Headquarters, 1916 Race St., Philadelphia 3, Pa.

* Presented at the Fifty-Third Annual Meeting of the Society, June 26–30, 1950.

¹ Director of Building Materials Research, John B. Pierce Foundation, Raritan, N. J.

² F. O. Anderegg, "Testing Surface Waterproofers," *ASTM BULLETIN*, No. 156, January, 1949, p. 71 (TP 33). References to previous literature may be found in that paper.

The report of the American Concrete Institute Committee 616 on "Recommended Practice for the Application of Portland-Cement Paint to Concrete Surfaces," *Journal, Am. Concrete Inst.*, Vol. 21, No. 1, September, 1949; *Proceedings*, Vol. 46, p. 1, has bearing on cement type waterproofers.

materials, including several new types, have been under test since that report. As a result a classification is proposed to cover their effectiveness in waterproofing, being based on the 72 hr. absorption per square foot under a $\frac{1}{4}$ -in. head of water:

- Grade A, below 0.2 lb.
Grade B, between 0.2 and 0.5 lb.
Grade C, between 0.5 and 1 lb.
Grade D, over 1 lb.

This classification affords a fairer means of grading waterproofing materials. Pending further investigation, this system of classification may serve for general use.

In addition, using the methods pre-

very difficult to estimate. The results reported here were obtained with side-cut, stiff-mud bricks obtained from the lower part of the kiln. Those having some pore openings ranging from $\frac{1}{16}$ to $\frac{1}{8}$ in. were selected. When it was desired to note the ability of a given material to close the openings, bricks were chosen having cracks ranging up to $\frac{1}{16}$ in.

DISCUSSION OF WATERPROOFERS

Cement-Base Waterproofer:

These usually require rather hard manual effort to work them into the superficial pores at the wall surface, in order to replace all the air therein with

TABLE I.—EVALUATION OF SURFACE WATERPROOFERS.

G = Good; F = Fair; P = Poor.

Identification Number	Absorption Class	Transpiration	Resistance to Efflorescence	Resistance to Weathering	Initial Appearance	Ease of Application	Cost of Application, cents per sq. ft.
CEMENT TYPE							
A.....	D	G	F	F	G White	F	8
B1.....	D	G	F	F	G White	F	8
B2.....	D						
C.....	D	G	F	P	G White	F G	8
D.....	D	G	F	F	G Dark brown	F	8
E.....	C	G	F	..	G White	F	8
F.....	D	G	F	..	G White	F	8
G.....	D	G	F	F	G Yellow	F G	8
H.....	A	G	F	G	G White	F G	8
I.....	D	G	P	P	Uneven	P	8
K.....	A	G	F G	..	G White	F G	8
L.....	A	G	F	G	G White	F	8
M.....	B	G	F G	P	G White	F	8
N.....	D	G	P	P	G White	F	10
O.....	C	G	F	F G	G White	F	8
SOLVENT TYPE—CLEAR							
A.....	D	F	F	F	Shiny	G	9
B.....	D	F	P	F G	Shiny	G	10
C.....	D	F	F	F	Slightly shiny	G	8
D.....	B	F			Shiny	G	9
E.....	D	P	P	P	Shiny	G	8
F.....	C	P			Shiny	G	9
G.....	D	F	P	P	Shiny	G	9
H.....	D	F	P G	P	Shiny	G	8
I.....	D	P	P	P	Shiny	G	9
K.....	D	P	P	P	Shiny	F Sticky	9
L.....	A	F	P	F	Shiny	G	10
SOLVENT TYPE—PIGMENTED							
A.....	D	F G	F	F	White	G	10
B.....	D	F	F	F	White	G	9
C.....	A	F	F	F G	White	G	10
D.....	B	F	F G	F	White	G	9
E.....	B	P	F	..	White	G	8
F.....	C	P	F	..	White	G	8
G.....	B	P	G	P	Gray	F G	8
EMULSION SYSTEMS							
A.....	B	G	F	F	Shiny	G	8
B.....	D	G	F	F	Shiny	G	8
C.....	B	G	F	F	Shiny	G	8
D.....	D	G	F	F	Shiny	G	8
E.....	B	G	Shiny	G	8
F.....	B	G	Shiny	G	8
G.....	D	F G	P	..	Shiny	G	10
SILICONES							
A.....	A	G	G	G	None	G	12
B.....	D	G	G	G	None	G	9
C.....	A	G	G	G	None	G	8
D.....	A	G	None	G	9
E.....	C	G	None	G	9
SPRAYED-ON STUCCOS							
A.....	B	G	F	F G	White	F	10
B.....	B	G	F	F	White	F	10
C.....	A	G	G	G	Glossy white	G	10

the suspension. The suction of the base must be brought under control by proper wetting before making the application of most of the materials of this class while curing by proper wetting, especially in summer, is very important.

All four grades are represented in the cement-base materials tested, while the observed, fairly rapid transpiration rates of all tested are similar to those giving satisfactory service in the field. The best of these have resisted efflorescent action quite well, but almost all have suffered on exposure on the roof over the winter in the presence of sodium sulfate. Some scale off as shown in Fig. 1. Others of low cement content may be eroded as in Fig. 2, while others are self-chalking and continue to present a fairly clean surface (Fig. 3). Typical absorption data are found in Fig. 4.

Solvent Type Waterproofer:

It is not difficult to formulate solvent type systems, especially when pigments are present, which will seal pores in masonry up to about $\frac{1}{16}$ in. or even up to $\frac{1}{4}$ in. by increasing pigment concentration. Linseed oil has been painted on concrete surfaces for many years with rather good results. One of the limitations of vegetable oil vehicles lies in the danger of saponification by alkalies found so commonly in portland-cement structures.

But many oleoresins have excellent resistance to chemicals and light. Typical results for water ingress are included in Fig. 5. Most of this type belong to grade A or B when freshly applied, but tend to degenerate gradually due to the action of the various effects involved in weathering, including efflorescent crystal pressure, oxidation, and preferential wetting.

Many oleoresinous films are rather low in water vapor transmission; in other words, they do not "breathe" very well. Any accumulation of soluble salts at the surface on which they have been painted will tend to cause the formation of water blisters by osmotic pressure. Since soluble salts are nearly always to be found in masonry and since alternate wetting and drying will tend to cause their accumulation at the surface, trouble of this sort is to be anticipated. Figure 6 presents an example.[1]

In certain types of wall, however, interior ventilation by means of cavities which are vented to the outside, either accidentally or systematically, may take care of the moisture so that films of poor transpiration characteristics may yet render good service. Concrete masonry houses have been very successfully painted with certain oleoresinous paints. These paints have the advantages of

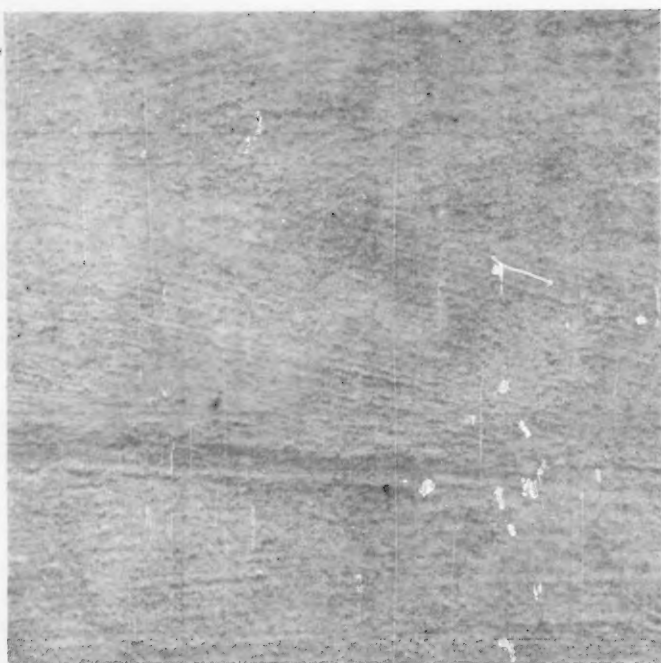


Fig. 3.—Slow Self-Chalking Has Kept This Surface Relatively Clean.

ease of application and good appearance. With the proper material, excellent durability can be secured in the paint film itself, when freed from the exfoliating forces of growing crystals or water blisters.

Emulsion Type Waterproofer:

A number of resins, usually with plasticizers, such as polyvinyl acetate and polystyrene, are readily obtained as emulsions in water. They can generally be applied with ease to masonry, no pre-wetting or aftercuring being required. Some effort should be made to work them into the larger pores, and appropriate concentrations should be used. According to our studies, rather thin films, suitably plasticized to give a good degree of flexibility, are capable of giving good service.

Of course, if a driving rain should strike the wall before the films have had a chance to dry out, the films may be washed off. Some of the dried films have a slight tendency to "blush" or turn white when wetted, but soon become colorless on drying again. This property of blushing seems to diminish with age. Pigments help fill larger openings, but naturally change the appearance of the wall.

Emulsion films seem to be characterized by good breathing ability. They cannot be expected to withstand the forces exerted by efflorescent crystal growth. The results obtained with the bricks, confirmed by field experience, show that these materials can be applied to the inside of a cellar wall to prevent moisture oozing through, but will not resist water coming out of larger open-

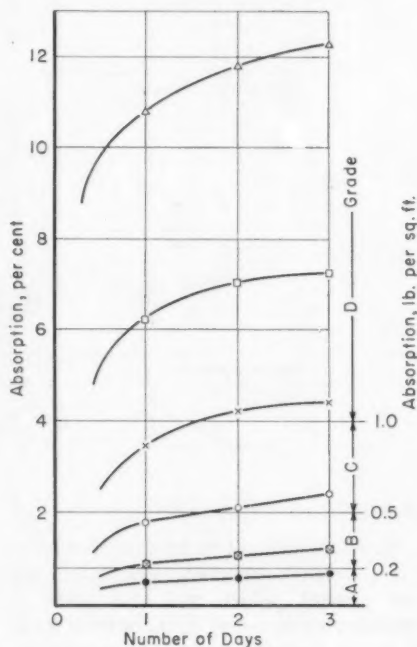


Fig. 4.—Cement Base.

ings under some pressure. Such places must first be filled with mortar or with a plugging cement. Figure 7 charts typical absorptions of this type of waterproofer. Among the emulsions tested, all four grades have been represented.

Silicones:

Silicones have been gaining prominence for a wide variety of applications, including masonry waterproofing. The silicone film produces less change in the appearance than any other waterproofer tried. One manufacturer recommends flooding on the surface; but as a dilute solution is used, the surfaces

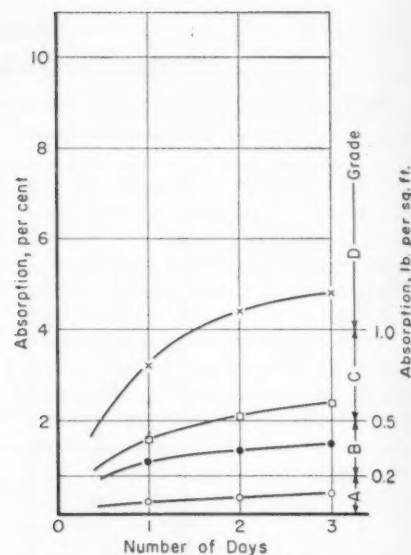


Fig. 5.—Solvent Systems.

of the pores are probably lined only a few molecules deep. It is believed that a chemical bond is formed with silica molecules in the masonry, so that it may be necessary to confine the application to silicates, which include brick, mortar, concrete blocks, sand-lime brick, etc.

The hydrocarbon part of the silicone compounds is water repellent so that the pores have negative capillarity and resist the ingress of water. However, water under higher pressure will pass into the larger openings, and anything greater than about $\frac{1}{16}$ in. should first be pointed up. Figure 7 also gives some absorption results. The silicones tested are classed as grade A. Silicones do not seem to interfere with breathing to any appreciable extent, an important property.

Bricks treated on five sides were placed with the untreated sides down in



Fig. 6.—Moisture and Soluble Salts Tend to Play Havoc with Solvent-Type Surface Treatments on Masonry.

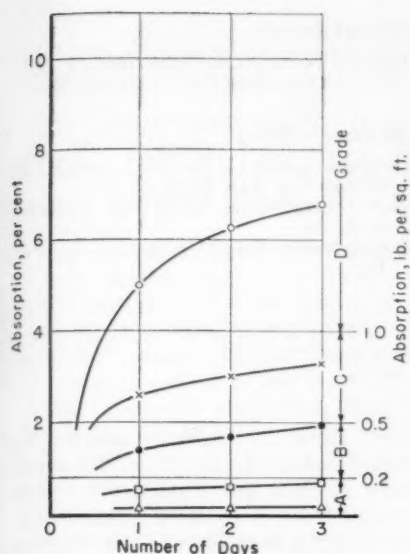


Fig. 7.—Emulsions and Silicones.

Upper of our curves were obtained with emulsions, while the bottom curve represents the maximum obtained in these experiments with silicones.

10 per cent sodium sulfate solution, but showed no appearance of salt in a week. When placed on the roof through the last part of the winter of 1949, especially during March, no evidence of harm could be detected. A year later, however, the treated sides of some bricks were spalled off to the depth of about $\frac{1}{4}$ in., probably due to freezing while saturated, and much of the water repellency had disappeared.

Stucco Waterproofing:

A great many million square yards of cement block walls have been coated with stucco applied by hand, and when reasonably good materials and workmanship are employed good results have generally been obtained. Such ready-mixed materials as "California Stucco" have given good service through the years. However, application costs are rather high, so that attention has lately been turned to spraying. Three types of spraying equipment have been studied in this investigation:

1. The materials mixed dry are placed in a hopper and drawn by a Venturi effect to the nozzle where the necessary water is added. Air under high pressure is required.

2. A fine sand and a cement containing a stearate integral waterproofer and a little lime are mixed with just enough water to form a soft plastic mass which is introduced into a small hopper above a spray gun. The gun operates at about 15 psi. pressure.

3. The solids are suspended in a lot of water, are forced under pressure through a long hose to the spray nozzle where air under considerable pressure atomizes the suspension.

Cinder block piers have been rendered tight by all three methods, at least after

preliminary soaking periods. The second method is preferred as being most nearly foolproof, and both laboratory tests and field observation indicate that it is probably most capable of providing the best results. Figure 8 shows piers coated by the second and third method.

One limitation of spraying is that a rough surface is produced, much like stippling. Such a texture can be used to very good advantage over rough block masonry. It can be smoothed out with a small amount of effort, when a plane finish is desired. Another limitation lies in the spatter, similar to that produced in paint spraying. However, the speed of covering is sufficient to give spraying of thin coatings some economic advantage. All three methods can be applied to give grade A results.

The sprayed-on stuccos seem to permit adequate breathing. The stucco applied by the second method and containing stearate waterproofer has resisted the tendency of sodium sulfate solution to form an efflorescent deposit in the laboratory but has not yet been tested on the roof. This material and method have been developed in connection with a cement block system which is laid up "dry wall." The sprayed-on stucco serves to fill the joints, apparently far better than in orthodox walls laid up by most masons.

CONCLUSIONS

The choice among these materials will be governed by a variety of considerations, such as: cost and availability of material, cost of application, type and age of wall to be treated, its exposure, opportunity for internal ventilation of the wall, etc. Each has its advantages and each its limitations.

In order to summarize factors influencing the applicability of different types of waterproofer to particular cases, the following chart is presented. This chart gives the results of dozens of individual tests on various brands and types of waterproofer. The chart, of course, is useful only as a general guide because such things as "cost" and "ease of application" depend on factors which can only be comparative under one set of conditions. It is believed, nevertheless, that this chart will be of value when used with discretion.

Cost estimates for two-coat applications on masonry of average roughness have been made, based on these experiments. For cement type waterproofer about eight to ten cents a square foot, which allows for material and conscientious scrubbing in, should be figured. For sprayed-on stucco these two items will run to about ten to twelve cents. For all of the solvent and emulsion systems the costs will be as



Fig. 8.—Sprayed-On Stucco Panels.

low as eight cents and as high as twelve cents, depending upon the material cost. Pointing up openings too large to be sealed may run the cost up another three to five cents. This item is avoided where stucco is sprayed on, however.

Acknowledgment:

Grateful acknowledgment is made to Hans Sternheim for assisting in obtaining the results reported here.

MATERIALS

The materials covered included cement-type waterproofer, colored solvent systems, pigmented solvent systems, and clear and pigmented emulsions. The materials were supplied by the individual companies as indicated below and grateful acknowledgment is made to the suppliers of these materials:

Cement-Type Waterproofer:

Abbey Hart Co., E. Orange, N. J.
 Alexite Engineering, Colorado Springs, Colo.
 American StaDri Corp., Brentwood, Md.
 Celadri Corp.
 Haynes Products Co., Omaha, Nebr.
 Industrial Plastics Co., Jacksonville, Fla.
 International Aquella Products Co., New York, N. Y.
 Kay-ite Co., W. Orange, N. J.
 Medusa Portland Cement Co., Cleveland, Ohio
 Reardon Co., St. Louis, Mo.
 Riverton Lime and Stone Co., Riverton, Va.
 Sika Chemical Co., Passaic, N. J.
 Standard Dri Wall Products, New Eagle, Pa.

Solvent Systems, Colorless:

Brooklyn Varnish Co., Brooklyn, N. Y.

Dewatex Manufacturing Co., New York, N. Y.
 Dewey & Almy Chemical Co., Cambridge, Mass.
 Du Pont Company, Fabrics and Finishes Division, Philadelphia, Pa.
 Haynes Products Co., Omaha, Nebr.
 Sika Chemical Co., Passaic, N. J.
 Stanley Chemical Co., E. Berlin, Conn.
 Stringer, H. R., River Road, Bound Brook, N. J.
 Xpando Corp., Long Island City, N. Y.

Solvent Systems, Pigmented:
 American Pipe and Construction Co., Los Angeles, Calif.
 B. F. Goodrich Chemical Co., Cleveland, Ohio
 Truscon Laboratories, Detroit, Mich.
 Shawinigan Products Co., New York, N. Y.
Emulsions, Clear and Pigmented:
 Bakelite Corp., New York, N. Y.
 Du Pont Co., Electrochemicals Division, Wilmington, Del.

Silicone Resins:
 General Electric Co., Waterford, N. Y.
 Wurdack Chemical Co., St. Louis, Mo.
Sprayed-On Stucco
 Bondact Cement Gun Co., c/o R. E. Brooks Co., New York, N. Y.
 Norris Self-O-Line Block Co., Philadelphia, Pa.
 Pennsylvania Spray-Crete Co., Chester, Pa.

DISCUSSION

MR. CARL A. MENZEL (*presented in written form*).¹—In its practical implications it occurred to me that a description of details of the method of applying the different types of waterproofing would give the reader a better idea of how the results reported could be realized in service. I note that under the heading "Cement-Base Waterproof" the initial sentence reads as follows:

"These (Cement-Base Waterproofers) usually require rather hard manual effort to work them into the superficial pores at the wall surface, in order to replace all the air therein with the suspension. . ."

I am glad that Mr. Anderegg made it clear that work had to be done in the case of portland-cement-base paints, and we advocate scrubbing them in for best results. Similarly, a description of the recommended practice for applying other surface waterproofing would be helpful.

MR. F. O. ANDEREGG (*author*).—The method of application was covered in the earlier paper.² A scrubbing is essential for good results with the cement type waterproofers. It takes a lot of "elbow grease," as we pointed out in the first paper. This has also been covered in an article appearing in *Better Homes and Gardens*, September, 1948.

As for the others, they can be applied with a paint brush. Those who handle silicones prefer to flood them on in the hope that by flooding only one coat will be needed. However, somewhat better results would be obtained with silicone in two coats.

These other materials require two coats, except for the sprayed-on stucco, where one application is sufficient. The cost of all these materials is about the same per square foot while the cost of

application is greatest, I think, with the cement type, because of the extra work.

The sprayed-on stuccos are rapidly applied comparing in cost with the application of the paint type waterproofers.

MR. R. S. WISHART.³—Lately we have been interested in the silicone field, and have made some "range-finding" tests on the use of silicones as masonry waterproofers. While we can not claim to have anything so complete as Mr. Anderegg's information, we have noticed a number of points in the use of these materials.

One of them is that the flooding application is essential for the proper performance of a silicone masonry water repellent, and we say water repellent, rather than waterproof.

Mr. Anderegg has made a good point in saying that two coats are better than one. Using his mode of testing with a similar brick, we have found in general that two coats give a superior performance. One coat usually performs quite as well as the conventional materials with which we have compared the silicone.

The penetration which seems important in the effectiveness arises from an adsorption process; that is, the solvent in which the silicone is embodied will penetrate to a greater or lesser degree, depending upon the porosity of the material. But it will penetrate, in general, about four or five times as far as the zone of the water repellency penetrates.

In other words, we believe there is an adsorption of the silicone from the solvent, and it should depend upon the specific surface area of the masonry involved.

Concerning the resistance to efflorescence of the silicones, we have run efflorescence tests on specially prepared mortar blocks treated with our and other silicones and with commercial aluminum stearate and drying oil masonry water repellents. The blocks were subjected to a 10 per cent sodium sulfate solution tests, the treated faces of the blocks being out of the solution and attacked from below by the solu-

tion, rising by capillary action. We found that the drying oil and the aluminum stearate failed relatively rapidly within 6 hr. and 24 hr., respectively, after having been placed in the solution. The silicones had lasted 168 hr. when the test was halted.

MR. F. J. MAIDULIER.⁴—I should like to ask Mr. Anderegg what are the "breathing properties" of these silicones.

MR. ANDEREGG.—The transpiration of the silicone is comparable to that of untreated brick. The pores have not been filled. They have simply been lined apparently with a very thin membrane, possibly three or four molecules deep, or a very thin film, so their area has been very little reduced.

MR. WILLIAM M. SPURGEON.⁵—One interesting thing that Mr. Wishart mentioned was that the solvent appears to penetrate more than the silicone does. I think that is probably due to the fact that the silicone is removed by chemical reaction with adsorbed water on the surface of the pores. Silicate material—perhaps I should limit that to glass—exposed to ordinary air, is covered with a layer of adsorbed water about 300 molecules thick, and if the silicone in this manner reacts with adsorbed water, molecule for molecule, it takes a lot of silicone to use up that adsorbed water before it gets down to the surface of the brick, and makes the relatively permanent bond which is supposed to be formed there. I think this may account for the fact that the silicone is "unavailable" after it has penetrated to any appreciable extent.

Mr. Anderegg mentioned that the concentration of silicone used was 1 per cent. I would like to know if this value is what the Linde Co. regards as commonly acceptable for a surface type waterproofer based on silicone. Silicones are quite expensive, and 1 to 2 per cent, as I recall, is about all that can be put in a solution which can still compete price-wise with those colorless waterproofers which are a solution of paraffin, say in mineral spirits.

I should also like to ask Mr. Anderegg whether it is his opinion that the service

¹ Manager, Housing and Cement Products Bureau, Portland Cement Assn., Chicago, Ill.

² F. O. Anderegg, "Testing Surface Waterproofers," *ASTM BULLETIN*, No. 156, January, 1949, p. 71 (TP 33).

³ Laboratory, The Linde Air Products Co., Tonawanda, N. Y.

⁴ Field Engineer, Dewey & Almy Chemical Co., Cambridge, Mass.

⁵ Research Director, American Fluresit Co., Inc., Cincinnati, Ohio.

life of the silicones is of the order of one to two years.

MR. ANDEREGG.—About the adsorption, Mr. Spurgeon is probably right. There may be primary or secondary valence chemical reactions with some adsorption.

But suppose one treats a material that is not siliceous, such as a limestone. That needs to be explored more fully.

In reply to the second question—the service life will depend upon the severity of the exposure. The bricks described in the paper were exposed on the roof, where the exposure is severe. But if they are protected by overhanging roofs, much longer life is to be anticipated.

If silicone waterproofers are applied to side walls exposed to storms off the ocean, their life would be somewhat shortened; while on application to parapet walls or penthouses on such a building on the ocean front an effective life of not more than two years can be expected. On the unexposed side of this same building effective service of, say, ten years can be predicted.

MR. NOLAN D. MITCHELL.⁶—Did Mr. Anderegg conduct any experiments on

⁶ Chief, Fire Protection Section, National Bureau of Standards, Washington, D. C.

⁷ Anderegg, Pfeffer, Judy, and Huber in "The Efflorescence and Staining of Indiana Limestone," *Purdue Univ. Eng. Expt. Sta. Bull.* 33, 1928, describe the exfoliation and disintegration limestone, sandstone, brick, and concrete masonry.

renewing the waterproofing after severe exposures? If so, was the coating more effective in the renewed state than originally?

MR. ANDEREGG.—Very few experiments of this type have been made. When soluble salts are present, successful waterproofing is extremely difficult.

MR. SPURGEON.—Were the silicone types tested of the silicone varnish type or of the type that react chemically with water and are supposed to make a permanent bond with the masonry?

MR. ANDEREGG.—I believe that the type that reacts with water was used in the tests.

MR. WISHART.—I may be able to contribute something on that point. Apparently our tests have paralleled Mr. Anderegg's, independently, and covered more silicones and probably fewer waterproofers. On the question which was originally raised by Mr. Spurgeon about the reactivity of the silicones with the water, I am not so sure that that reaction does take place. Also, the silicones under consideration are dissimilar to glass in structure despite several advertising claims to the contrary. Although the literature indicates relatively little difference in water repellency for films laid down by various organosilicon monomers, the silicone polymers do vary widely in repellency.

MR. ANDEREGG.—An interesting phenomenon has developed since this paper was written. Some of the bricks treated with a silicone waterproofer were placed with the untreated side in contact with a 10 per cent solution of sodium sulfate, so that a considerable amount of salt was drawn up into the bricks. These bricks were exposed during the first four months of 1950 on the roof, during which time the waterproofed parts of the brick were spalled off. The forces involved were those developed by growing crystals of sodium sulfate, which could be plainly seen.⁷ The magnitude of these forces is of the order of 100,000 psi., a value so great that no surface treatment can be expected to stand up very long in the presence of soluble salts and moisture. These forces are causing constant deterioration of our masonry structure. The only way to prevent such action is to keep moisture out of the walls.

A sprayed-on stucco, waterproofed with stearate, suffered a similar fate under this exposure. In other experiments, five out of the ten bricks used in testing different waterproofers were filled with sodium sulfate and exposed on the roof. The other five bricks were free from sodium sulfate and exposed as controls in the same way. The latter are generally in good condition while the former have lost a large part of their coatings, pushed off by crystal growth.

Testing Cement Base Paints and Dampproofers¹

By William M. Spurgeon²

SYNOPSIS

Tests made on 23 commercial cement paints, 21 of which are used for dampproofing masonry walls, show that they vary widely in porosity (water absorptions range from 2.4 to 65.1 per cent by weight) and 7-day compressive strengths (which range from 0 to 3300 psi.). In general, for comparable curing conditions, high absorption values accompanied low compressive strengths and *vice versa*.

The theory of dampproofing is discussed briefly, and the need for further development of a simple, short screening test to supplement weathering data for cement-base dampproofers is shown. The need for establishing specifications for these materials is also pointed out.

EVERY year hundreds of thousands of dollars are spent for cement-base paints,³ used for decorating

NOTE.—DISCUSSION OF THIS PAPER IS INVITED either for publication or for the attention of the author. Address all communications to A. S. T. M. Headquarters, 1916 Race St., Philadelphia 3, Pa.

¹ Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

² Research Director, American Fluoresit Co., Inc., Cincinnati, Ohio.

³ Statistical Supplement to Weekly Letter, National Paint, Varnish and Lacquer Association, Inc., Washington, D. C., July 21, 1947.

concrete and masonry walls and generally, also, either for keeping water out of such walls or at least for preventing its passage through the walls.^{4 5 6} The commercially available cement paints

⁴ C. C. Fishburn and D. E. Parsons, "Tests of Cement-Water Paints and Other Waterproofings for Unit-Masonry Walls," *Report BMS 95*, Nat. Bureau of Standards (1943).

⁵ Standard Specifications for Hollow Load-Bearing Concrete Masonry Units (C 90-44), 1949 Book of ASTM Standards, Part 3, p. 626.

⁶ F. O. Anderegg, "Testing Surface Water Proofers," *ASTM BULLETIN*, No. 156, January, 1949, p. 71 (TP33).

are known to vary widely in both durability and effectiveness as water barriers. It is desirable, therefore, to have tests and specifications for these coating materials.

The ASTM has issued no such specifications, the only ones available at present being Federal Specification TTP-21⁷ and A.C.I. Standard 616-49.⁸ The first gives certain requirements as to composition and methods of examination. The second discusses composition, manufacture, storage, method of application, and general characteristics, including durability. However, neither one gives a method of determining how effective a cement paint of unknown composition is likely to be as a dampproofer, nor is any simple

⁷ Federal Specification for Paint, Cement-Water, Powder, White and Tints (for Interior and Exterior Use), *Federal Specification TTP-21* (1941).

⁸ G. E. Burnett, F. O. Anderegg, et al., "Proposed Recommended Practice for the Application of Portland Cement Paint on Concrete Surfaces," *Journal, Am. Concrete Inst.*, Vol. 21, No. 1, September, 1949; *Proceedings*, Vol. 46, pp. 1-16.

method given for judging its durability. A consideration of dampproofing theory and the properties of portland cement is useful for suggesting an ideal cement-base dampproofer with which commercial coatings can be compared, especially in regard to water resistance and service life.

DAMPPROOFING AND THE LAWS OF CAPILLARY FLOW

A distinction is made here between waterproofing materials, used on walls subject to considerable hydrostatic pressure, and dampproofing materials, used on walls subject to little or no hydrostatic pressure and taking in water mainly by capillarity.

The importance of the capillary forces for saturation of porous concrete or masonry construction has, of course, been emphasized many times, on the bases of experimental work^{9, 10} and also calculations involving the laws of capillary rise and viscous flow. A combination of these laws has been effected by a number of workers,^{11, 12, 13} giving equations describing the dynamics of capillary flow:

$$l^2 = \frac{\gamma \cos \theta}{2\eta} rt, \dots \dots (1)^{14}$$

$$Q = \frac{v}{t} = \frac{\pi r^2 \gamma \cos \theta}{4\eta l} \dots \dots (2)^{14}$$

In these equations, l is the length of capillary of radius r , filled in time t by a liquid of surface tension γ (as determined by a dynamic method) and viscosity η . The contact angle between the capillary walls and the liquid is θ , and Q is the volume v of liquid flowing into the tube in t seconds.

In deriving these equations, it is assumed that (1) the liquid flow is laminar, rather than turbulent; (2) the contact angle is less than 90 deg.; (3) one end of the capillary is open to the air, so that the gas is not trapped and compressed by the oncoming liquid; (4) the resistance to air flow through the capillary and out of its end is negligible; (5) the capillary tube is horizontal so that gravitational effects can be neglected, and there is no hydrostatic pressure acting on the liquid in the tube; (6) the radius r is sufficiently large so

that molecular or ionic fields do not extend across the tube. The justification for the first of these assumptions has been given by Washburn.¹¹ For ceramic and other siliceous construction materials, θ would undoubtedly be less than 90 deg. and most probably could be taken as 0 deg. The validity of the assumption regarding entrapment of air is not so certain if the equations are to be applied to masonry materials.¹⁵ This point will be discussed later in connection with the experimental work. The fourth assumption was also justified by Washburn.¹¹ The fifth assumption, of course, is made for the purpose of simplifying the equations and focusing attention on the capillary flow, uncomplicated by other factors. In regard to the sixth assumption, it appears that our knowledge of pore size and pore size distribution in masonry materials is meager. For certain types of bricks a "mean effective capillary radius" of the order of 1 to 5 microns has been given.¹⁶ However, much larger holes and cracks, visible to the unaided eye, are often found in bricks. For the densest possible hardened portland-cement paste, Powers and Brownyard¹⁷ have estimated the average pore to be from 20 to 40 Å in diameter. Any movement of water through such small pores must be more like diffusion than flow due to pressure or capillary forces. By far the greatest part of the flow through mortar or concrete, however, must occur not through these pores but rather through the much larger inhomogeneities or channels that form in the paste and under the coarse aggregate particles during bleeding or that are left as a result of improper blending of ingredients, or through cracks that form after the material has hardened.

The direct application of Eqs. 1 and 2 to the problems of water flow through porous media has been criticized for various reasons.¹⁸ For example, the presence of enlargements in the otherwise narrow tube may stop capillary flow. Again, the presence of water may cause swelling, with consequent change in the capillary size. Still, the equations will be applicable to any given portion of a capillary where conditions may be considered uniform, and qualitative use of the equations will be of value in understanding what must be done to achieve dampproofing.

¹⁵ Longitudinal absorption of certain liquids by wood is known to be greatly increased if one end of the specimen is left dry and exposed to air.

¹⁶ R. T. Stull and P. V. Johnson, "Some Properties of the Pore System in Bricks and Their Relation to Frost Action," *Journal of Research*, Nat. Bureau of Standards, Vol. 25, No. 6, December, 1940, p. 722 (RP1349).

¹⁷ T. C. Powers and T. L. Brownyard, "Studies of the Physical Properties of Hardened Portland Cement—Part 3—Theoretical Interpretation of Adsorption Data," *Journal, Am. Concrete Inst.*, Vol. 18, No. 4, December, 1946; *Proceedings*, Vol. 43, p. 497.

Inspection of Eq. 2 shows that use can be made of three variables, r , θ , and l , in developing a masonry dampproofing coating based on portland cement. The most important of these variables is r , occurring as it does to the third power. If the radii (and hence the volumes) of all the capillaries in the coating be minimized, then the amount of water passing through the coating will be greatly reduced. The capillary volumes at a given age will be measured, roughly, at least, by the total amount of water absorbed by the material when saturation is attained. It follows that such a coating with high water absorption contains a large volume of capillary spaces through which water can pass.¹⁸

The second variable, θ , can be used to advantage in two ways. First, one can incorporate water-repellent materials integrally in the coating. Such surface-active substances should be dispersed as uniformly as possible throughout the coating material, as by ball milling or, preferably, by incorporating the waterproofing ingredients in such form that the actual water-repellent material is formed by chemical reaction just after the mixing water is added.

The third variable, l , should also be made use of if maximum dampproofing is to be attained. In ordinary language this means that high coverage should be sacrificed, and that a distinction should be made between a portland-cement paint, which is ordinarily applied in a very thin, porous layer, and a portland-cement dampproofing material applied by brushing but in a relatively thick layer.

With the aid of these considerations, it is now possible to set up requirements for an ideal dampproofing coating—based on cement and applied by brushing. First, it should bond strongly to masonry, with some dampproofing achieved by a kind of silting or pore plugging process. Second, it should harden rapidly after application to prevent damage by rain or freezing. Third, it should have negligible porosity (low total water absorption), and what few pores there are should be extremely small, that is, the coating material should have a slow rate of water absorption. Fourth, it should be difficult to wet after it is on the wall. Fifth, it should be applicable in a thick layer. Sixth, it should show good durability under weathering. In addition it should have certain qualities desirable in any water paint, such as easy mixing with water, good brushability, high hiding power, both wet and dry, and freedom

¹⁸ It must be borne in mind that two similar materials can have equal water absorption values, and yet one, with a few large capillaries, may offer much less resistance to passage of water than the other, with many small capillaries.

⁹ J. M. Bell and F. K. Cameron, "The Flow of Liquids Through Capillary Spaces," *The Journal of Physical Chemistry*, Vol. 10, p. 659 (1906).

¹⁰ W. M. Dunagen, "Methods for Measuring the Passage of Water Through Concretes," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, pp. 866-880 (1939).

¹¹ E. W. Washburn, "Dynamics of Capillary Flow," *The Physical Review*, Vol. 17, pp. 273-283 (1921).

¹² E. K. Rideal, "On the Flow of Liquids Under Capillary Pressure," *The Philosophical Magazine*, Vol. 44, pp. 1152-1159 (1922).

¹³ J. J. Bikerman, "Surface Chemistry for Industrial Research," Academic Press, Inc., New York, N. Y., pp. 20-27 (1948).

¹⁴ It may be mentioned in passing that these equations are useful in developing the colorless, transparent types of waterproofing liquids.

from efflorescence. It should be slow chalking to give a surface that is clean but does not wear away too fast, and should not be easily soiled, but have good washability. We are concerned here mainly with the requirements of extremely low water absorption and durability. We want a combination of ingredients, a method of blending and storing them, and a method of application that will give a thick coating with capillaries as few, as small, and as water-repellent as possible. From a knowledge of cement characteristics we would expect such a finish to be dense and strong and correspondingly durable. As Committee 616 of the American Concrete Institute has pointed out, "considering the nature of portland cement paint it appears that durability is dependent upon the strength, hardness, and density of the paint film."¹⁸

It is not to be expected that such a dampproofing material can be developed that will have zero porosity. Nevertheless it is of interest to find out to what extent various materials now on the market approach this ideal. The simple water absorption tests described below were made for this purpose. They serve to screen cement coatings of low porosity, for further testing, from those of high porosity. Also described is a simple method, based on 7-day compressive strengths, of judging whether the cement-water ratios are high enough to insure good durability.

MATERIALS

Twenty-three commercial cement base paints and a simple sand-cement wash were used for these tests. Twenty-one of the paints were variously described as dampproofers. Eight of the products were packaged in cardboard cartons or in paper bags at the time of purchase. The others were packaged in metal containers.

TEST METHODS

The paints, mixed with water in accordance with the manufacturers' directions, were cast in uncoiled 2-in. cube molds for the absorption tests and in 2 by 4-in. cylinder molds for the compression tests. No correction was made for bleeding. Storage was in the laboratory air (65 to 80 F.), unmolded after 24 hr. (except for a few exceptionally fragile specimens which had to be left in molds for a longer period). Absorption tests were begun at 144 hr. after water was added to the powder. Compression tests were run at 168 hr., application of pressure being as prescribed in ASTM Method C 39-49.¹⁹

¹⁸ Standard Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39-49), 1949 Book of ASTM Standards, Part 3, p. 830.

For the absorption tests the cubes were placed in water at 65 to 85 F. in such a way that only the top surfaces of the cubes remained exposed to air. In general, three specimens were used for both tests, and averages were computed, the per cent absorption being calculated by weight.

It was realized that the cubes were probably not completely free of evaporable water at the age of 6 days, and that the cement hydration reactions were not complete at that age. Acceleration of drying by heating, however, was not considered desirable, partly because of the effect of heat on cement hydration and partly because of the possibility of lime-silica reaction. Prolonged air drying was therefore resorted to as being more like what the coatings are subjected to in service. On completion of the absorption tests at age 6 days, the cubes were allowed to air dry at about 75 F. for 18 weeks, after which the tests were repeated.

TABLE I.—ABSORPTION AND COMPRESSIVE STRENGTH DATA FOR 24 CEMENT-BASE COATINGS.

Product	Water-Solid Ratio, qt. per 5 lb.	Absorption at Age 6 days, per cent	Absorption at Age 19 weeks, per cent	7-day Compressive Strength, psi.
A.....	1.00	2.19	2.4	1320
B.....	1.00	1.8	2.73	2790
C.....	1.00	2.02	2.9	1430 ^a
D.....	1.00	4.80	...	2015
E.....	0.96	10.2	5.82	>1910
F.....	1.00	7.27	5.87	414
G.....	1.00	2.72	6.7	3300
H.....	0.75	11.0	15.1	1680
I.....	1.00	12.2	...	611
J.....	1.11	20.4	21.0	900
K.....	2.28	34.2	22.4	345
L.....	2.00	19.4	27.0	1123
M.....	1.50	30.6	30.3	34 ^c
N.....	1.88	22.8	33.9	840
O.....	1.88	16.7 ^d	35.1	522 ^d
P.....	1.60	44.3	40.8	0 ^e
Q.....	1.79	12.0 ^f	41.2	1070 ^f
R.....	2.00	36.7	44.8	140
S.....	2.0	24.4	45.4	410
T.....	2.50	46.1	50.8	48
U.....	h	39.7	52.6	215
V.....	2.50	55.2	...	223
W.....	2.75	60.8	...	87
X.....	2.25	29.6	65.1 ⁱ	315

^a Two cylinders only.
^b Cement wash, 1 lb. cement, 2.75 lb. standard Ottawa sand.
^c One cylinder only.
^d Two days in mold.
^e One specimen only.
^f Cured in mold; absorption test at 5 days.
^g 1 to 1 by volume.
^h 5 to 4 by volume.
ⁱ One cube only.

DISCUSSION

Results of this survey of properties are shown in Table I. For most of the cubes the adsorption curves, which were of typical shape, leveled off within a few hours. The 24-hr. absorption values recorded, therefore, are very close to the saturation values. Some of the low-absorption cubes, however, continued to gain weight very slowly, even after 260 hr. of immersion. For the high-ab-

sorption cubes, in general, the rates of absorption were also high.

It is fortunate that a rough measure of the porosity is all that is needed for these products, for the change in weight of the cubes on immersion is the result of a number of effects: (1) absorption of water into the pores (assumed to be by far the major effect), (2) continuing hydration of the cements, (3) leaching out of lime and soluble salts, (4) carbonation of lime in the blocks, and (5) chipping of corners and edges, inevitable in handling the more fragile specimens. In spite of these effects, the results were quite reproducible. Individual 24-hr. absorption values for product L are typical in this respect: 26.5, 27.8, and 27.1 per cent. Only two of the paints showed wide variations of individual values from the average: individual values for product O were 13.1, 15.6, and 21.4 per cent, and for product K were 31.5, 34.1, and 37.2 per cent.

The compressive strength data are also to be regarded as rough, partly because of the variation in curing conditions and partly because it was difficult to handle the more fragile cylinders without chipping their edges. Nevertheless, these data afford an indication of the cement content of the paint, at least when paints having the same water-powder ratio are compared. Though other factors also enter, such as carbonation of free lime, presence of retarders or accelerators, or use of high early strength cements, the strengths nevertheless give a means of screening out those paints with too little cement to be durable on weathering.⁸ The strongest cubes were also the hardest, in general.

It may perhaps be claimed that this method of testing cement-base coatings is not satisfactory because the materials spread out in film form might be expected to show properties different from those shown by the same product in 2-in. cubes. For example, unless the film is very thoroughly cured, more complete hydration of the cement is to be expected in the cubes. Yet it must be remembered that if the cubes of a given product are very porous, the less thoroughly hydrated material in the film must be still more porous. It might also be objected that the absorption of a 2-in. cube gives no exact measure of the porosity of a cement paint containing much free lime, since little allowance is made for the effects of carbonation—a process that certainly occurs to a considerable extent in the film. Since the density of calcium hydroxide is 2.24 and that of calcium carbonate is 2.72, the carbonation process must result in reduced porosity. It has been shown, however, that it is difficult to justify

the inclusion of lime in a cement-base coating. It is certainly not needed for good brushability or for making the coating resistant to passage of water. It undoubtedly weakens the film.⁸ In fact, such lime-containing films can range in composition all the way from whitewashes hardened with a little cement to cement paints extended—or adulterated—with a little lime. The chief justification for inclusion of lime in a cement paint is, apparently, the resultant saving in manufacturing cost.

There is no question of the desirability of measuring the effectiveness of cement-base dampproofers by applying them as a thin layer to the surface of some kind of reproducible concrete, mortar or brick, then determining the rate of absorption of the total unit at various ages. Because dampproofing effectiveness is a function of coating thickness, however, it would be necessary to have a method of applying films of uniform dry thickness, identical for each coating. Such a method has not been developed for cement-base coatings, and this is the main reason why such a test procedure was not adopted. In any case, it would be more useful for research than as a quick means of judging field performance of these coatings.

In view of the porous nature of masonry or concrete walls, it must be realized that a certain degree of "waterproofing" can be achieved with such a simple substance as a mixture of finely ground silica or lime and water brushed on the wall, with no binder whatsoever. The brushing forces the solid particles into the larger openings, where they become wedged and partially block the flow of water. None of the particles, of course, would adhere to the outside surface of the wall once the water evaporated. In other words, the coating would dust badly. Such a mixture represents one extreme of the possible brush type dampproofers. At the other extreme, we have well-formulated coatings containing an ample quantity of binder which penetrates into the pore openings on brushing, giving the pore plugging type of dampproofing; such coatings also cover the outer surface of the wall with an adherent layer of low

porosity—a layer that acts as a barrier to keep water out of the wall. The word "layer" is used here in preference to "film," for since no material was found that has zero porosity, it follows from the laws of capillary flow that when dampproofing is desired, coverage should be sacrificed in order to build up as thick a coat as will not crack on subsequent aging or weathering. Naturally, a product that has high porosity, as evidenced by high absorption, and which gives an extremely thin film of high coverage, cannot be expected to achieve dampproofing except in the very mildest cases of masonry dampness. This is borne out by observation, after rains, of cement block buildings coated with some of the high-absorption paints.

In view of the results given above it is suggested that a distinction be made between cement-base coatings that accomplish dampproofing and those that are merely decorative. Such a distinction would have to be somewhat arbitrary. It is evident that absorption values lower than 5 per cent can be attained, however, and this would be one possible dividing point.

There is one other point of interest in regard to the results given above. The principal destructive agent for a cement-base coating is water, which acts in at least two ways: (1) freezing and expanding in the pores, causing the film to flake off, and (2) dissolving the binder and eroding away the film. A hard, insoluble layer with low porosity and high strength will naturally be able to resist these effects better than a soft, relatively soluble film of high porosity and low strength. As shown in Table I, low porosity and high (compressive) strength generally go together (for comparable curing conditions), though there is no exact relationship.

If absorption and strength tests are to be used for research purposes or even for setting up specifications it is desirable that certain improvements be made in the procedures. Mixing and curing conditions should be more exactly defined for both the absorption and the compression tests, for example, for the compression tests the water-powder ratio used should be the same for all

paints. For the absorption test the drying conditions should be more reproducible; the test should be started after cement hydration is reasonably complete and the drying curve for the specimen has leveled off. Possibly the drying processes could be hastened and the drying and carbonation processes made more like what actually occurs when the paint is put on a masonry wall by using thin disks rather than cubes for the absorption test. Closer control of the temperature during the entire course of the test would also be desirable.

SUMMARY

The laws of the dynamics of capillary flow indicate that for maximum effectiveness a cement-base dampproofing coating should be thick, with water repellent pores as few and as small as possible.

A rough measure of the porosity of such coatings is given by their water absorption values, which for the commercial coatings tested ranged from 2.4 to 65.1 per cent by weight at age 19 weeks.

This spread of values suggests the desirability of distinguishing between cement paints which merely decorate and have high porosity, and cement dampproofers which have low porosity.

Seven-day compressive strengths for the coatings tested ranged from 0 to 3300 psi. Since publications from other laboratories indicate that the durability of a cement coating is dependent on its cement content—the greater the percentage of cement, the greater the durability—it would appear that the weaker coatings, at least, can hardly be very durable.

In view of these wide variations of properties and the large quantities of these coatings sold for dampproofing purposes, it is evident that specifications might well be set up for them.

Acknowledgment:

The tests described in this paper were made with the assistance of Mrs. Ila Carlson, John Watt, Charles Tidd, and Jerry Orndorff.

DISCUSSION

the masonry are brought near the surface and deposited there behind the dampproofing material, with the crystals growing in oriented directions and pushing off the surface treatment.

Mr. W. M. SPURGEON (*author*).—Around Cincinnati we have not often encountered that condition, even in

basements. However, efflorescent crystal pressure must certainly be added to the list of destructive agents for cement-base paints and dampproofers. Have you observed it mostly on concrete blocks or bricks?

Mr. ANDEREGG.—Anywhere on masonry building materials of a porous

Mr. F. O. ANDEREGG.¹—With reference to disintegration reactions, Mr. Spurgeon should not forget efflorescent crystal pressure. The soluble salts in

¹ Director of Building Materials Research, John B. Pierce Foundation, Raritan, N. J.

character. I have seen it all over this country, and in Europe. .

MR. SPURGEON.—I have seen such coatings pushed off porous walls subject to, say, an 8-ft. head of water. There

is no doubt that the efflorescent crystal pressure can be of even greater magnitude. The solution for this problem, of course, is prevention: (1) soluble salts, or materials that form them, must be

kept out of masonry materials during their manufacture; (2) entry of such salts from outside should be eliminated by using preventive rather than remedial waterproofing.

Interpretation of Tension Creep-Time Relations

By Joseph Marin¹ and L. W. Hu¹

THE evaluation of creep strain is particularly important in the design of machine and structural parts operating at elevated temperatures. Excessive creep deformations during the life of a part may make the part unserviceable. Many empirical relations have been used to determine the relation between the creep strains or creep strain rates and stress.²

The proposed equations for the creep-stress relation that give good agreement with test results are too complicated to use. Furthermore, unless a simple creep-stress relation is used for simple tension, the derived creep stress and strain relations for bending, torsion, and combined stresses become so complicated that it is impractical to use them. The purpose of this paper is to propose a creep-stress relation for creep of metals at elevated temperatures which is simple to use and at the same time gives a good fit to the test data.

PROPOSED CREEP-STRESS RELATION

A creep strain-time relation for a given stress value is shown in Fig. 1. An examination of this creep-time plot shows that there are three stages in this relation. There is first a primary stage where the creep rate or slope of the creep-time curve decreases. This is followed by a secondary stage during which the creep rate or slope of the creep-time curve remains essentially constant. Finally, there is a tertiary stage where the creep rate gradually increases until rupture occurs. For stress values that would be used in design, the tertiary stage of creep is not present and it is only necessary to consider the primary and secondary stages of creep. In the past, creep-stress relations have been used where the primary stage has been neglected. It is then assumed that the initial creep strain *OA* (Fig. 1)

compared to the total creep strain is small. This assumption is a satisfactory one if the time considered is large. The commonly used log-log relation makes this assumption since it is assumed that the creep rate-stress relation is given by the equation

$$C = BS^n \dots \dots \dots (1)$$

where:

C = the constant creep rate,
S = the tensile stress, and
B and *n* = experimental constants.

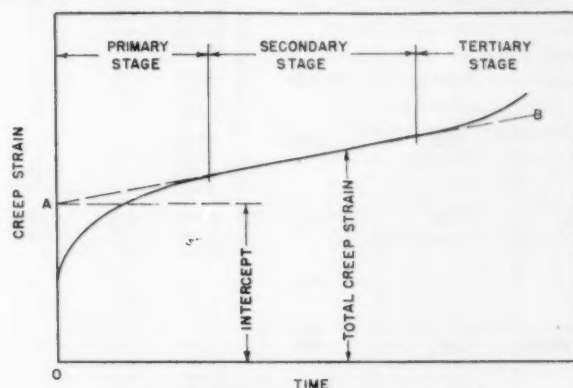


Fig. 1—Creep Strain-Time Relation.

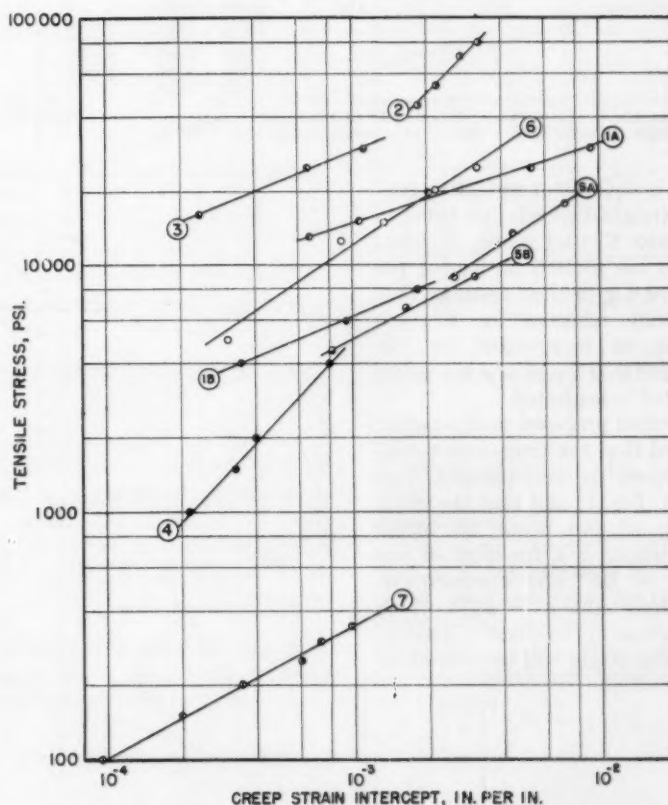


Fig. 2—Variation Between Creep Strain Intercept and Stress.

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¹ Professor of Engineering Mechanics and Research Professor of Engineering Materials, and Research Assistant, respectively, The Pennsylvania State College, State College, Pa.

² J. Marin, "Mechanical Properties of Materials and Design," McGraw-Hill Book Co., Inc., New York, N. Y. (1942).

TABLE I.—COMPOSITION OF MATERIALS.

	Composition, per cent												Brinell Hardness	Lab.
	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Nickel	Chromium	Tungsten	Vanadium	Molybdenum	Copper	Zinc		
No. 1a....	0.15	0.50	0.025	0.032	0.23	123	3a
No. 1b....	0.15	0.50	0.025	0.032	0.23
No. 2....	0.48	0.49	0.016	0.015	0.62	...	1.20	0.52	285	3b
No. 3....	0.15	0.24	0.009	0.022	0.12	0.04	1.34	0.68	145	14
No. 4....	0.45	0.47	0.014	0.015	0.77	...	1.40	0.98	0.25	285	3
No. 5a....	2.44	0.76	0.26	0.057	1.11	16.56	3.30	7.30	...	195 to 215	12e
No. 5b....	2.44	0.76	0.26	0.057	1.11	16.56	3.30	7.30
No. 6....	0.69	20.00	74.23	5.08	74 ^a	20f
No. 7....	Pb alloy 2P (0.03 per cent calcium)												...	21w

^a 1000-kg load.

TABLE II.—EXPERIMENTAL CREEP CONSTANTS.

No.	Material	Page Number in References ^a	Temperature, deg. Fahr.	Stress, psi.	Time, hr.	Creep Rate, in. per in. per hr.	Intercept, in. per in.	B	n	D	m
No. 1a...	Steel	20-21	800	13 000 15 000 20 000 25 000 30 000	992 1015 1000 999 1021	0.00 × 10 ⁻⁶ 0.00 0.16 0.70 2.10	0.67 × 10 ⁻² 1.06 2.02 5.30 9.12	7.23 × 10 ⁻¹⁵	6.36	9.42 × 10 ⁻¹⁷	3.12
No. 1b...	1000	4 000 6 000 8 000	1004 1003 1005	0.36 1.13 2.93	0.35 0.94 1.84	5.01 × 10 ⁻¹⁸	3.01	8.17 × 10 ⁻¹⁸	2.40
No. 2....	Steel	190-191	800	45 000 53 800 70 000 80 000	500 500 500 500	0.09 0.15 0.49 0.20	1.84 2.21 2.76 3.27	5.07 × 10 ⁻¹⁷	2.00	5.46 × 10 ⁻⁸	0.97
No. 3....	Steel	304	850	16 000 25 000 30 000	1500 1500 1500	0.08 0.12 0.16	0.24 0.66 1.11	2.58 × 10 ⁻¹²	1.06	1.80 × 10 ⁻¹⁴	2.40
No. 4....	Steel	492	1200	1 000 1 500 2 000 4 000	500 500 500 500	1.86 2.26 3.82 31.30	0.21 0.33 0.40 0.80	5.50 × 10 ⁻¹³	2.12	3.39 × 10 ⁻⁷	0.93
No. 5a....	Cast iron	712	842	8 960 13 440 17 920	1000 1000 1000	0.46 0.92 1.9	2.58 4.45 7.2	4.42 × 10 ⁻¹⁸	2.02	3.87 × 10 ⁻⁹	1.47
No. 5b....	1000	4 480 6 720 8 960	1000 1000 1000	0.13 0.21 1.05	0.82 1.64 3.25	2.87 × 10 ⁻¹⁸	2.89	5.17 × 10 ⁻¹¹	1.96
No. 6....	Copper alloy	741	600	5 000 12 500 15 000 20 000 25 000	1000 1000 1000 1000 1000	0.00 0.06 0.13 0.34 0.72	0.31 0.90 1.34 2.15 3.20	1.94 × 10 ⁻²²	3.54	1.36 × 10 ⁻⁹	1.43
No. 7....	Lead alloy	833	110	100 150 200 250 300 350	2000 2000 2000 2000 2000 2000	0.010 0.050 0.060 0.200 0.360 0.510	0.09 0.20 0.35 0.61 0.73 0.97	6.11 × 10 ⁻¹⁵	3.11	1.62 × 10 ⁻⁸	1.88

^a The page numbers refer to "Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys," Am. Soc. Testing Mats. and Am. Soc. Mechanical Engrs., March, 1938. (Issued as separate publication, STP No. 37.)

Equation 1 is equivalent to stating that there is a straight-line relation between the creep rate, C , and stress, S , when these values are plotted on log-log paper. The log-log method assumes that the creep-time relations for a given stress value as represented by the straight line AB (Fig. 1) and the intercept strain OA is neglected.

In the method proposed in this paper, it is assumed that the creep-stress relation is replaced by two straight lines OA and AB (Fig. 1) and that the creep is made up of two parts—an initial strain OA which is a function of the stress or $e' = DS^m$ and a subsequent time-dependent value $e'' = BS^n t$, which also is a function of the stress. That is, the total creep strain will be defined by

$$e = e' + e''$$

or

$$e = DS^m + BS^n t \dots (2)$$

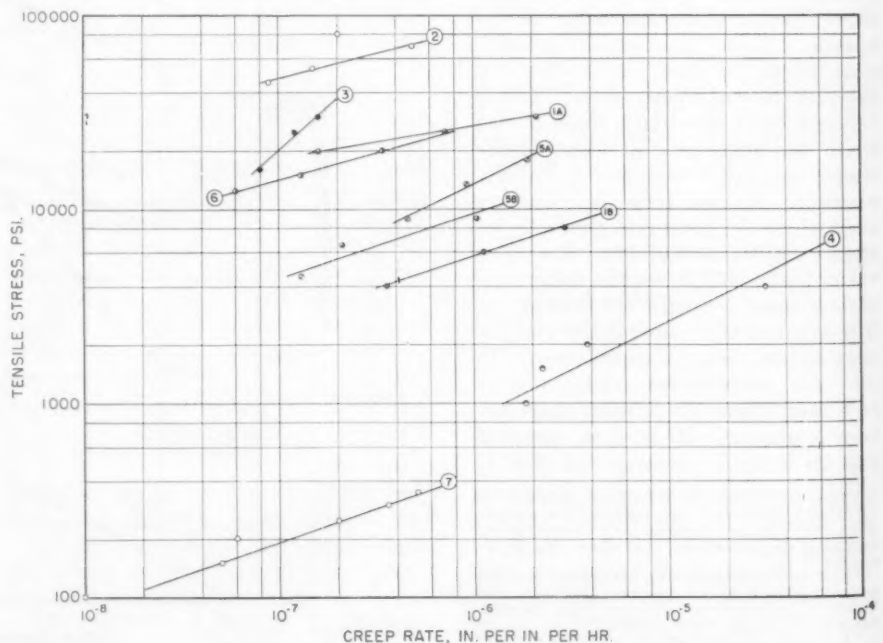


Fig. 3—Relation Between Creep Rate and Stress.

TABLE III.—COMPARISON OF EXPERIMENTAL AND THEORETICAL CREEP STRAIN.

Lab.		Stress, psi.	Time, hr.	Total Creep, in. per in.		Difference, per cent
				Experimental	Theoretical	
3a	No. 1a.....	13 000	992	0.68×10^{-2}	0.65×10^{-2}	+4
		15 000	1015	1.05	1.02	+2
		20 000	1000	2.16	2.68	-19
		25 000	999	6.12	5.64	+8
		30 000	1021	11.26	10.92	+3
20f	No. 1b.....	4 000	1004	0.72	0.71	+2
		6 000	1003	2.04	2.05	0
		8 000	1005	4.90	4.71	+4
21w	No. 2.....	45 000	500	1.87	1.89	-1
		53 800	500	2.28	2.26	+1
		70 000	500	2.97	2.85	+4
		80 000	500	3.37	3.38	0
3.12	No. 3.....	16 000	1000	0.33	0.31	+5
		25 000	1000	0.72	0.81	-11
		30 000	1000	1.21	1.22	-1
2.40	No. 4.....	1 000	500	1.15	0.85	+35
		1 500	500	1.45	1.81	-19
		2 000	500	2.30	3.17	-27
		4 000	500	17.00	12.80	+32
0.97	No. 5a.....	8 960	1000	3.04	2.98	+2
		13 440	1000	5.37	5.62	-4
		17 920	1000	9.10	8.85	+3
2.40	No. 5b.....	4 480	1000	0.95	0.90	+5
		6 720	1000	1.85	2.12	-13
		8 960	1000	4.3	3.91	+10
0.93	No. 6.....	5 000	1000	0.33	0.28	+14
		12 500	1000	0.95	1.13	-16
		15 000	1000	1.49	1.51	-2
		20 000	1000	2.46	2.44	+1
		25 000	1000	3.92	3.64	+7
1.47	No. 7.....	100	1000	0.10	0.10	-2
		150	1000	0.25	0.24	+3
		200	1000	0.41	0.44	-8
		250	1000	0.82	0.72	+13
		300	1000	1.09	1.08	+1
1.96		350	1000	1.46	1.53	-5

where:

D and m = constants determined from the creep intercept-stress relation, and

B and n = creep constants obtained from the creep rate-stress relation $C = BS^n$ since $C = \dot{\epsilon}''/t$ or $\dot{\epsilon}'' = BS^n t$.

This paper presents the results of a number of tension creep investigations to show that Eq. 2 is a good approximation.

COMPARISON OF PROPOSED CREEP-STRESS RELATION WITH TEST DATA

In comparing the proposed method of interpretation with test data, creep test results for seven different materials were selected.³ The materials selected, their composition, and the laboratory designation are listed in Table I. Table II shows the values of the stresses, temperatures, and creep strains for each material.

Figure 2 shows the relation between the creep strain intercept and stresses

³ "Compilation of Available High-Temperature Creep Characteristics of Metals and Alloys," Am. Soc. Testing Mats., and Am. Soc. Mechanical Engrs., March, 1938. (Issued as separate publication, STP No. 37.)

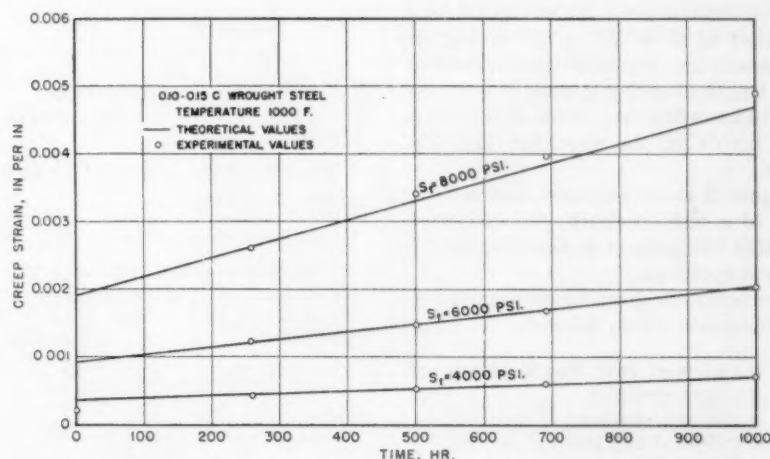


Fig. 4—Comparison of Theoretical and Experimental Creep Strain Values.

for each material. The straight lines drawn in Fig. 2 for each material were selected using the method of least squares. Values of the constants D and m based on the straight lines $\dot{\epsilon}'' = DS^m$, as shown in Fig. 2, are listed in Table II.

The creep rate constants B and n used to represent the time-dependent parts of the creep $\dot{\epsilon}'' = BS^n t$ or $C = \dot{\epsilon}''/t =$

in the development of a mechanics of creep.

Acknowledgment:

The study reported in this paper is part of an investigation on the mechanics of creep sponsored by the Research Corp. The cooperation and financial assistance of the Research Corp. in making this research possible is greatly appreciated.

An Evaluation of the Effective Gage-Length Equivalent of the Fillet and Shoulder of the Gage Length Portion of a Tension Test Bar Under Creep and Stress-Rupture Conditions¹

By H. V. Kinsey²

SYNOPSIS

The errors introduced into creep measurements when the over-all test specimen (gage length, fillets, shoulder) are included in the extensometer system are examined. Three high-temperature alloys are used as examples. It is shown that it is not possible to arrive at a common correction factor for the equivalent gage length, even for one alloy, if the true effective gage length is to be known within limits of ± 1 per cent, unless a test specimen having an 8-in. gage length is employed.

VARIOUS means of attaching extensometer reference strips to a tension test specimen to obtain creep measurements at elevated temperature were considered. One of the suggested methods measured, in effect, the total creep taking place over all of the bar between the threaded ends, that is, the parallel gage-length section plus both fillets at the ends of the gage length plus both shoulders. This length is represented as $G + 2F + 2S$ in Fig. 1. To determine the equivalent effective gage length of such a system, it is necessary to know the magnitude of the creep that occurs in the shoulders and the fillets.

Figure 2 is an enlarged sketch of a fillet of a test specimen, the radius of the fillet being equal to the diameter of the test specimen.

The notations given below are used in the discussion which follows:

- R_0 = radius of gage length portion of test specimen,
- R_s = radius of shoulder of test bar,
- R = radius at any point P in fillet,
- r = fillet radius,
- h = vertical distance from end of gage length portion A to P ,
- $x = R - R_0$
- σ = stress in bar at distance h above A ,
- σ_1 = stress in gage length portion of bar,
- σ_0 = constant,
- v_0 = constant, and
- v = creep rate, per cent per hour.

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¹ Published by permission of the Director-General of Scientific Services, Department of Mines and Technical Surveys, Ottawa, Canada.
² Head, High Temperature Metals Section, Physical Metallurgy Division, Mines Branch, Department of Mines and Technical Surveys, Ottawa, Canada.

CALCULATIONS OF CREEP IN THE FILLET

In order to calculate the creep and creep rate in the fillet of the test specimen, it was found expedient to consider increments of the fillet in terms of increments of R .

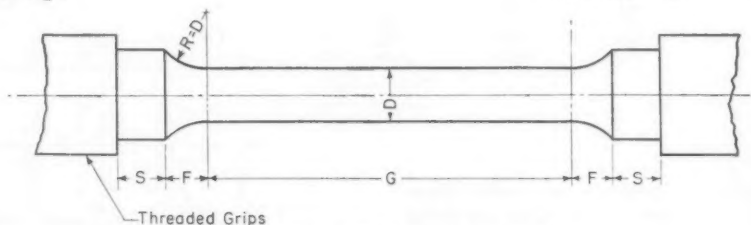


Fig. 1.—Typical Test Specimen Design.

Knowing R_0 , R , and r , h may be determined as follows:

$$\begin{aligned} h^2 &= r^2 - (r - x)^2 \\ &= 2rx - x^2 \\ \therefore h &= \sqrt{4R_0(R - R_0) - (R - R_0)^2} \quad (\text{see Fig. 2}) \\ &= \sqrt{6RR_0 - 5R_0^2 - R^2} \dots \dots (1) \end{aligned}$$

and

$$\sigma = \sigma_1 \frac{R_0^2}{R^2} \dots \dots (2)$$

According to A. Nadai and P. G. McVetty,³ the relationship between stress and creep rate are given by the expression

$$v = v_0 \sinh \frac{\sigma}{\sigma_0} \dots \dots (3)$$

Substituting for σ from Eq. 2 in Eq. 3, this becomes

$$v = v_0 \sinh \frac{\sigma_1 R_0^2}{\sigma_0 R^2} \dots \dots (4)$$

Using the relationships in Eqs. 1 and

³ A. Nadai and P. G. McVetty, "Hyperbolic Sine Chart for Estimating Working Stresses of Alloys at Elevated Temperatures," *Proceedings, Am. Soc. Testing Mats.*, Vol. 43, pp. 738-748. (1943).

2, fillet creep in a standard $\frac{1}{2}$ -in. diameter bar for Inconel X, alloy S-816, and alloy 735 was calculated for fixed conditions of stress and temperature, as outlined below.

In using the relationships given in Eqs. 1 and 4, certain constants (R_0 , R , and σ_1) can be fixed from consideration of the test specimen dimensions and of the stress in the gage length; other constants (v_0 and σ_0) are dependent on the material and the temperature of testing.

For the test specimen under consideration,

$$R_0 = 0.25 \text{ in.}$$

$$R_s = 0.31 \text{ in.}$$

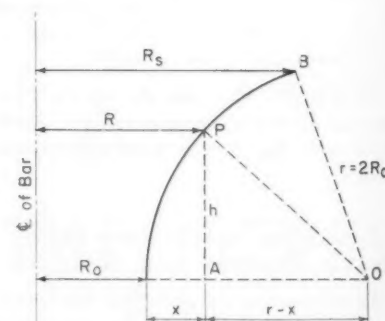


Fig. 2.—Test Specimen Fillet.

Substituting in Eq. 1 for R_0

$$h = \sqrt{R(1.5 - R) - 0.3125}$$

Inconel X at 1500 F.:

From data published by the International Nickel Co., Inc.,⁴ the creep rate for Inconel X at 1500 F. and for given stresses is

Stress, psi.	Creep Rate, per cent per hr.
20 000.....	2.3×10^{-4}
15 000.....	1.8×10^{-4}

⁴ Inconel "X" Data Sheet, January, 1947, Development and Research Division, The International Nickel Co., Inc., New York 5, N. Y.

TABLE I.—CALCULATION OF FILLET CREEP IN INCHES FOR INCONEL X AT 1500 F. AND 20,000 PSI.

R	h	Δh	R^2	$\frac{0.0846}{R^2}$	$\sinh \frac{0.0846}{R^2}$	$v \times 10^{-4}$	Average $v \times 10^{-4}$	100-hr. Fillet Creep 10^{-4}
0.250	0	0	0.06250	1.354	1.8073	2.260		
0.2505	0.0223	0.0223	0.06275	1.348	1.7950	2.245	5.252	5.020
0.251	0.0316	0.0093	0.06300	1.342	1.7827	2.228	2.236	2.080
0.252	0.0447	0.0131	0.06350	1.332	1.7623	2.202	2.215	2.903
0.253	0.0548	0.0101	0.06401	1.321	1.7401	2.177	2.190	2.212
0.254	0.0632	0.0084	0.06452	1.311	1.7202	2.150	2.164	1.818
0.255	0.0707	0.0065	0.06503	1.301	1.7004	2.125	2.137	1.387
0.260	0.0995	0.0288	0.06760	1.251	1.6038	2.005	2.115	6.090
0.265	0.1217	0.0222	0.07022	1.205	1.5185	1.900	1.953	4.335
0.270	0.1400	0.0183	0.07290	1.161	1.4400	1.801	1.851	3.390
0.275	0.1562	0.0162	0.07563	1.118	1.3659	1.708	1.754	2.840
0.280	0.1706	0.0144	0.07840	1.079	1.3009	1.626	1.667	2.402
0.285	0.1838	0.0132	0.08123	1.041	1.2395	1.550	1.588	2.098
0.290	0.1960	0.0122	0.08410	1.006	1.1845	1.480	1.515	1.848
0.295	0.2074	0.0114	0.08700	0.973	1.1340	1.417	1.449	1.647
0.300	0.2177	0.0103	0.09000	0.940	1.0847	1.356	1.386	1.427
0.305	0.2278	0.0101	0.09301	0.910	1.0409	1.310	1.333	1.346
0.310	0.2375	0.0097	0.09610	0.881	0.9995	1.250	1.280	1.254

Total creep in one fillet in 100 hr. = 44.097×10^{-4} in.
Total creep in both fillets in 100 hr. = 88.194×10^{-4} in.

By using these values in Eq. 3, values for v_0 and σ_0 may be obtained by graphical solution.³

At 1500 F., the values of Inconel X are:

$$v_0 = 1.25 \times 10^{-4}$$

$$\sigma_0 = 14,790 \text{ psi.}$$

Then

$$v = 1.25 \times 10^{-4} \sinh \frac{\sigma_1 \times 0.25^2}{14,790 R^2}$$

If

$$\sigma_1 = 20,000 \text{ psi.}$$

then

$$v = 1.25 \times 10^{-4} \sinh \frac{0.0846}{R^2}$$

The creep rate, v , is calculated for points P_1, P_2, P_3 , etc., where $R = R_1, R_2, R_3$, etc.

The amount of creep is then calculated for increments of length, Δh , between P_1 and P_2, P_2 and P_3 , etc., throughout the entire fillet, using the average of the creep rates for each two points considered. Typical calculations for Inconel X at 1500 F. and at a stress of 20,000 psi. are given in Table I.

It is shown in Table I that at 1500 F. under a stress of 20,000 psi., the total creep in both fillets in 100 hr. is 88.194×10^{-4} in. and the creep rate in the shoulder is 1.250×10^{-4} per cent per hr.

If the shoulder length at each end of the test bar is 0.25 in., then total shoulder length is 0.5 in. and total creep in both shoulders in 100 hr. is $1.25 \times 10^{-4} \times 0.5$ or 62.5×10^{-6} in. Therefore, total creep in both fillets and shoulders in 100 hr. is 150.69×10^{-6} in.

Gage-length creep rate = 2.260×10^{-4} per cent per hr., then gage length equivalent of shoulders plus fillets for Inconel X at 1500 F. and 20,000 psi. is

$$\frac{150.69 \times 10^{-6}}{2.26 \times 10^{-4}} = 0.667 \text{ in.}$$

In a similar manner the gage length

equivalent for Inconel X at 1500 F. and 10,000 psi. was found to be 0.542 in.

Alloy S-816 at 1350 F. and 1500 F.:

The calculations for fillet and shoulder creep of alloy S-816 at 1350 F. and 1500 F., using a standard $\frac{1}{2}$ -in. diameter test specimen and stresses as shown in Table II, were carried out in an identical manner to those for Inconel X.

These calculations are based on published values for creep rate of alloy S-816⁵ at the temperatures and stresses given below:

Stress, psi.	Creep Rate, per cent per hr.	
	1500 F.	1350 F.
20 000.....	0.003	0.00005
15 000.....		0.000025
10 000.....	0.00009	...

Values for v_0 and σ_0 for alloy S-816 at 1350 F. and 1500 F. were determined in the same manner as previously described for Inconel X. They are:

Temperature, deg. Fahr.	σ_0 , psi.	v_0
1350.....	7228	6×10^{-4}
1500.....	2838	5×10^{-4}

TABLE II.—ERRORS OF GAGE LENGTH BASED ON AVERAGE VALUES OF GAGE-LENGTH EQUIVALENT OF SHOULDERS AND FILLETS OF 0.456 IN. FOR CREEP-RUPTURE AND CREEP CONDITIONS.

Material	Stress, psi.	Tempera- ture, deg. Fahr.	Assumed Effective Gage Length, in.	True Effective Gage Length, in.	Error	
					Inches	Per cent
CREEP-RUPTURE CONDITIONS						
Alloy S-816.....	{ 40 000	1350	4	3.878	+0.122	+3.05
	{ 20 000	1500	4	3.813	+0.187	+4.7
Inconel X.....	20 000	1500	4	4.211	-0.211	-5.3
CREEP CONDITIONS						
Alloy S-816.....	{ 20 000	1350	4	4.069	-0.069	-1.7
	{ 10 000	1500	4	3.971	+0.029	+0.7
Inconel X.....	10 000	1500	4	4.086	-0.086	-2.2
Alloy 73J.....	20 000	1500	4	3.964	+0.036	+0.9

⁵ Technical Data on Alloy S-816, February 16, 1945, Research Department, Allegheny-Ludlum Steel Corp., Watervliet, N. Y.

⁶ Nicholas J. Grant, "The Cobalt-Chromium J Alloy at 1350 to 1800 F." Am. Soc. Metals, Preprint No. 17 (1947).

Gage length equivalents and the conditions for which they were calculated are as follows:

Stress, psi.	Gage-Length Equivalents of Fillets and Shoulders, in.	
	1350 F.	1500 F.
40 000.....	0.334	...
20 000.....	0.525	0.267
10 000.....	...	0.427

Alloy 73J at 1500 F.:

The creep in the fillets and shoulders of alloy 73J⁶ were calculated for a stress of 20,000 psi. at 1500 F. Those calculations are based on the stress and creep rate data given below:

Stress, psi.	Creep Rate, per cent per hr.
30 000.....	0.013
20 000.....	0.001

If these data are used in Eq. 3, then

$$v_0 = 1.4 \times 10^{-4}$$

and

$$\sigma_0 = 4000$$

The calculated gage-length equivalent of the shoulders and fillets for a standard

$\frac{1}{2}$ -in. diameter test specimen of alloy 73J at 1500 F. and 20,000 psi. is found to be 0.42 in.

Summary of Gage-Length Equivalents:

The calculated gage length that is equivalent in creep to the total shoulder plus fillet length of a standard $\frac{1}{2}$ -in. diameter test specimen under the various conditions and for the several alloys considered is as follows:

Stress, psi.	Gage-Length Equivalents, in.			
	S-816		Inconel X	Alloy 73J
	1350 F.	1500 F.	1500 F.	1500 F.
40 000.....	0.334		0.667	0.42
20 000.....	0.525	0.269	0.542	
10 000.....	...	0.427		...

DISCUSSION OF RESULTS

It is immediately apparent from an examination of the data summarized above that the gage-length equivalent is a specific value for each alloy at each stress and temperature, and that it would not be possible to design a bar that would have an accurately known effective gage length even for testing only one alloy at a specific temperature.

Let us examine the errors involved in attempting to design such a bar for use at 1350 F. and 1500 F.

The average value of all the gage-length equivalents given above is 0.456 in. The design of this specimen will be based on a gage-length diameter of 0.5 in., a shoulder diameter of 0.62 in., and a fillet radius of 0.5 in. The specimen is to have an effective gage length of 4 in.

Referring to Fig. 1:

Shoulder length, S	= 0.25 in.
Fillet length, F	= 0.238 in.
$S + F$	= 0.488 in.

TABLE III.—ERRORS OF GAGE LENGTH BASED ON GAGE-LENGTH EQUIVALENT OF SHOULDERS AND FILLETS OF 0.474 IN. FOR CREEP CONDITIONS.

Material	Stress, psi.	Tempera- ture, deg. Fahr.	Assumed Effective Gage Length, in.	True Effective Gage Length, in.	Error	
					Inches	Per cent
Alloy S-816.....	20 000	1350	4	4.051	-0.051	-1.3
	10 000	1500	4	3.953	+0.047	+1.2
Inconel X.....	10 000	1500	4	4.068	-0.068	-1.7
Alloy 73J.....	20 000	1500	4	3.946	+0.064	+1.6

length under the conditions given in Table II are +4.7 per cent and -5.3 per cent.

If, instead of using a value of 0.45 in. for the gage-length equivalent of the shoulders and fillets, the average of the equivalents for creep conditions, which is 0.474 in., is used, then the errors listed in Table III exist.

Therefore the limits of error of determination of effective gage length for creep conditions could not be expected to be less than ± 1.7 per cent, based on a 4-in. gage length. Since the true effective gage length should be known within limits of ± 1 per cent for accurate measurements of creep rate, a specimen having an effective gage length of 8 in. would be required in order to be certain that the gage-length error would be less than ± 1 per cent.

Since it is not always practical to use a test specimen having a gage length of 8 in., any extensometer system that measures over-all creep of the distance $(G + 2F)$ or $(G + 2F + 2S)$ as shown in Fig. 1 cannot be considered to have the required accuracy of effective gage length.

Satisfactory creep measurements can only be obtained by clamping the extensometer reference strips on to an accurately measured gage length of a test specimen which is uniform in cross-section throughout that gage length.

Total for both ends of bar
 $2(S + F) = 0.976$ in.
Gage-length equivalent of
 $2(S + F) = 0.456$ in.
Total effective gage length
required = 4 in.
Therefore parallel length, G
= 4-0.456
= 3.544 in.

The errors in such an assumption, for the materials, temperatures, and stresses under consideration may be divided into two general groups:

Group 1.—Conditions of stress and temperature which cause relatively short times to rupture, and relatively high creep rates, that is, creep-rupture conditions.

Group 2.—Conditions of stress and temperature which cause relatively long times to rupture and relatively low creep rates, that is, creep conditions.

The data for these two groups are given in Table II.

The maximum limits of error in this determination of true effective gage

Discussion of Paper on Stress Cracking of Polyethylene¹

MR. J. L. WILLIAMS.²—Did the author find any visual evidence of actual fractures? Polystyrene may show "craze" marks when tested in a non-solvent such as kerosine, and it is wondered if the lowered strain observed in polyethylene when tested in non-solvents might also result in cracks or other signs of surface attack.

MR. CAREY (author).—No, there was no visual evidence of crazing. Of course, the point where visual observation and microscopic observation merge is very hard to find.

MR. E. Y. WOLFORD.³—The results

¹ R. H. Carey, "Stress Cracking of Polyethylene," ASTM BULLETIN, No. 167, July, 1950, p. 56 (TP138).

² Laboratory Director, The Dow Chemical Co., Midland, Mich.

shown on the bar chart (Fig. 5), impress me with the similarity of your method of test to methods which have been proposed to test for unrelieved internal stresses. The proposals have been to bore a hole in the specimen and apply a nonsolvent. Particularly in polystyrene, observation is then made for the development of crazing or cracks. The plastic behavior appears to resemble, for instance, the well-known caustic embrittlement of steel. Hence, one might perhaps think that polystyrene also partakes of the "metal-like" characteristic of a plastic, particularly as polystyrene oriented by stretching while plastic exhibits elongation before failure

³ Chemical Div., Koppers Co., Inc., Monaca, Pa.

under tension test, that is, the fracture is "ductile" rather than "brittle."

In "as-molded" samples, the variation of strength consequent to change of visible or invisible crazing or cracking as varied with molecular weight can be ascribed to at least two variable factors: (1) the amount of unrelieved internal stresses due to the variable degree of chill of the specimen as molded, the chill varying with molecular weight because the solidifying temperature varies with molecular weight—call this the variation of intensity of stress with molecular weight, and (2) specimens of different molecular weight have better or poorer resistance to the same degree of unrelieved internal stresses because within limits, the physical properties improve

with increasing molecular weight—call this the variation of resistance to stress with molecular weight. It may be impossible to devise a test method which will evaluate the relative importance of either factor alone. However, it is possible to eliminate or at least minimize both factors simultaneously by annealing the specimen before test.

Hence, while from the presentation of the paper I suppose the specimens were tested in the "as-molded" condition, I should like to ask whether the specimens were annealed before testing.

MR. CAREY.—Mr. Wolford's question is concerned with the residual stresses induced by the molding, and whether I have any information on annealed specimens. My answer is no. I have no information on annealed specimens. I think it is probably true, however, that this so-called brittleness can be induced

by triaxial stress, with or without residual stresses.

MR. CAREY (*author's closure by letter*).—I would hesitate to compare this phenomenon in polyethylene with the crazing so often seen in polystyrene. The visual appearance of the surface of the polyethylene, located some distance away from the actual break, is very much like that of the original specimen. Polyethylene is much more opaque than polystyrene, however, and crazing cannot be so readily detected.

Polyethylene, like other crystalline plastics, shows the distinguishing characteristic of "necking" or drawing at the yield point. This yielding phenomenon is most easily analyzed from stress-strain curves obtained on constant rate of straining (crosshead travel) machines. The existence of an upper and lower yield point may be masked if the testing

machine is unable to follow the drop in load.

The "metal-like" characteristics of polyethylene are inferred from its behavior during room temperature stretching. The X-ray diffraction pattern of stretched polyethylene shows that the yield phenomenon is similar to the yield in metals. The crystallites align themselves approximately in the direction of the maximum shear stress. This inter-crystalline slipping or sliding may be inhibited by the geometrical form of the specimen. A hole was placed in the specimens primarily to inhibit failure by sliding or slip. This hole introduces triaxial stressing with a consequent reduction in the value of the shear stress. Residual stresses and methods of molding will affect the tail end of the stress-strain curve more than the initial part. This again is analogous to metals.

An Apparatus for Measuring Dielectric Strength at Elevated Temperatures¹

By W. Hand² and R. R. Winans²

SYNOPSIS

There is presented a description and details of construction of equipment for measuring the dielectric strength of insulating materials at elevated temperatures up to 250 C. The method described functions by use of silicone rubber gaskets in contact with the test specimen and under pressure to inhibit flashover. Data are presented for comparison of results obtained by this method with dielectric strength measurements in air and in transformer oil. It was observed that measurements with this device generally lie between measurements in air and in transformer oil.

THE need for plastic insulating materials capable of maintaining high dielectric strength at elevated temperatures has long been recognized. With this need has come the parallel requirement of developing methods for measuring the dielectric strength properties of materials at high temperatures. Such determinations may require equipment, materials, and techniques radically different from those now in use for measurements at ordinary temperatures.

The above problem was encountered in an investigation conducted at the Material Laboratory, New York Naval Shipyard. In this investigation, it was

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¹ The opinions or assertions contained herein are those of the authors and are not to be construed as reflecting the views of the Department of the Navy or the Naval Service at large.

² Technologist (Plastics) and Materials Engineer, respectively, Material Laboratory, New York Naval Shipyard, Brooklyn 1, N. Y.

necessary to determine the dielectric strength of several typical laminated thermosetting plastic materials at commercial power frequency and through a range of elevated temperatures up to 250 C.

The accepted methods³ available were found unsatisfactory for temperatures above 130 C. (flash point of transformer oil). Attempts to employ high-temperature insulating oils, such as silicone oil, chlorinated oils, etc., proved fruitless due to such factors as deterioration and change in properties of the oil with increased temperature, and the danger of toxicity and fire hazard. It was finally decided to develop equipment based on ideas furnished by the single-shot tester for dielectric strength test on insulating tape.⁴

³ Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (D 149-44), 1949 Book of ASTM Standards, Part 6, p. 411.

⁴ Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295-49 T) (Appendix), *Ibid.*, p. 299.

The principle upon which this equipment operates is that voltage flashover around the specimen may be greatly inhibited by the application of a sealing apparatus under pressure to the upper and lower faces of the test specimen, thus providing a dielectric barrier between the upper and lower electrodes. The material used for sealing must be flexible and sufficiently compressible to mold itself under pressure to the contours and other irregularities of the test specimens, and must also have dielectric strength high enough to cause dielectric breakdown to occur only in the specimen. For measurements in the vicinity of room temperature, ordinary non-conducting rubber is usually adequate for this purpose. For elevated temperatures up to 250 C., it was found that silicone rubber best meets these requirements. The electrode holders, high-voltage leads to the electrodes, and all equipment in the vicinity of the high voltage must be constructed of, or insulated by, materials that will successfully withstand the voltage stresses and will not deteriorate with prolonged exposure to the elevated temperatures.

CONSTRUCTION

The device developed for dielectric strength measurements at elevated temperatures is shown in Fig. 1. In this view may be seen both the thermally and electrically insulated high-

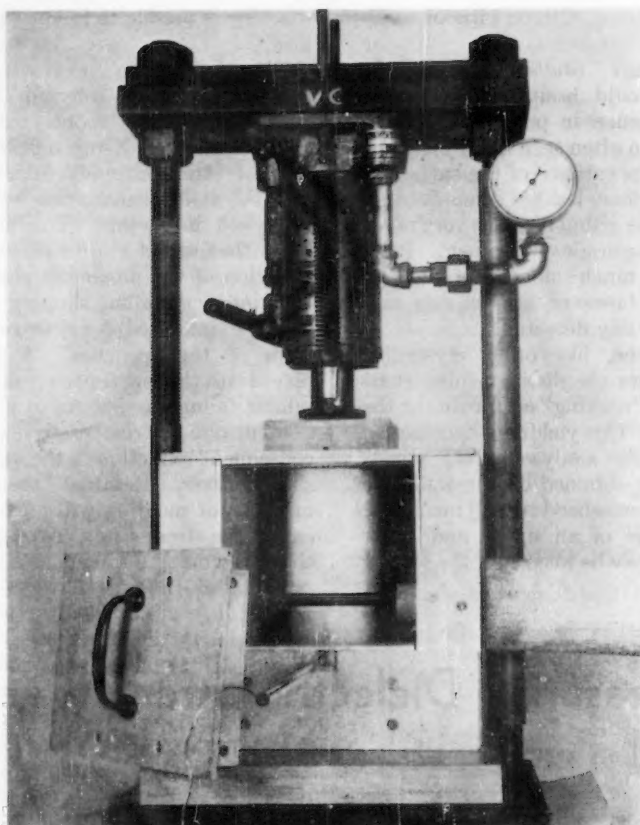


Fig. 1.—View of Dielectric Strength Measuring Device in High-Temperature Chamber.

temperature chamber enclosing the device and the thermally insulated tubes through which hot air, suitably controlled, is circulated in the chamber. The sealing apparatus used with this device was $\frac{3}{8}$ -in. thick silicone rubber gaskets cemented to the faces of the electrode mountings, using silicone rubber adhesive.⁵ Pressure on these gaskets was applied by operation of the hydraulic jack shown in the illustration. It was found that pressures of 150 to 250 psi. were sufficient for preventing flashover. The electrode holders are specially constructed cylindrical blocks of steatite⁶ with intersecting holes for the electrode assembly and for the test voltage lead-in, respectively. The lead-in as shown in Figs. 1 and 2 was insulated by $\frac{3}{8}$ -in. outside diameter capillary glass tubing. When used in the investigation, however, it was found that this type of insulation was too fragile for the mechanical operations involved and under high electrical stresses was subject to dielectric breakdown. "Kel-F"⁷ bars, $\frac{1}{2}$ by $\frac{3}{4}$ in., of suitable length were machined and substituted and found to be superior for this purpose. These lead-in insulators were sealed into the steatite electrode holders by the same

⁵ No. 12509 adhesive, General Electric Co.
⁶ Alsimag A-196, American Lava Co., Chattanooga, Tenn.
⁷ Monochlorotrifluoroethylene, M. W. Kellogg Co., Jersey City, N. J.

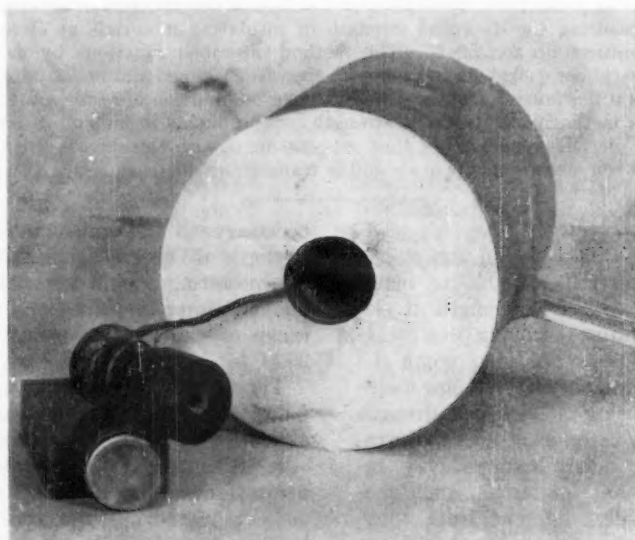


Fig. 2.—Disassembled Electrode and Mounting.

silicone adhesive used for cementing the silicone rubber gaskets. Tapped holes $\frac{1}{2}$ -in. deep in the base of the upper electrode holder permitted fastening to the hydraulic jack by means of a 4 by 4 by $1\frac{1}{2}$ -in. block of glass melamine laminated material press-fitted to the ram of the jack.

The electrode assembly is shown in Fig. 2. The electrodes ($\frac{3}{4}$ in. diameter

and rounded to $\frac{1}{8}$ -in. radius) are spring mounted and easily replaceable when excessively pitted. By mounting the electrodes on springs, the pressure exerted by the jack is not transmitted to the area of the test specimen covered by the electrodes. Alignment of the electrodes is accomplished by setting the base of the lower electrode holder in a collar of insulating material, aligning the electrodes by eye, and once aligned, bolting the collar firmly to the base plate.

The chamber enclosing the device is lined on the inside with $\frac{1}{8}$ -in. glass silicone laminated material and on the outside with $\frac{1}{4}$ -in. Transite. The space between the walls ($1\frac{1}{2}$ in.) is filled with fiber glass. Two 1-in. holes in one side of the chamber provide for the inlet and outlet hot-air ducts. A vertical slot in the rear wall provides for motion of the upper electrode lead-in insulator which is coupled to the ram of the jack. A feed-through hole is also provided in the front of the chamber below the removable door for the lower electrode lead-in insulator. A 4-in. square opening through the top of the chamber permits vertical motion of the upper electrode assembly for insertion and removal of test specimens. The block of melamine-glass cloth attached to the ram of the jack provides thermal insulation for the chamber as the ram

of the jack is elevated or depressed.

Control of the hot air entering the chamber is accomplished by a regulator operated by a thermocouple in the inlet duct. Experimentally it was determined that by maintaining the inlet air at a specific constant temperature, a stabilized temperature would in time be attained in the space between the electrodes.

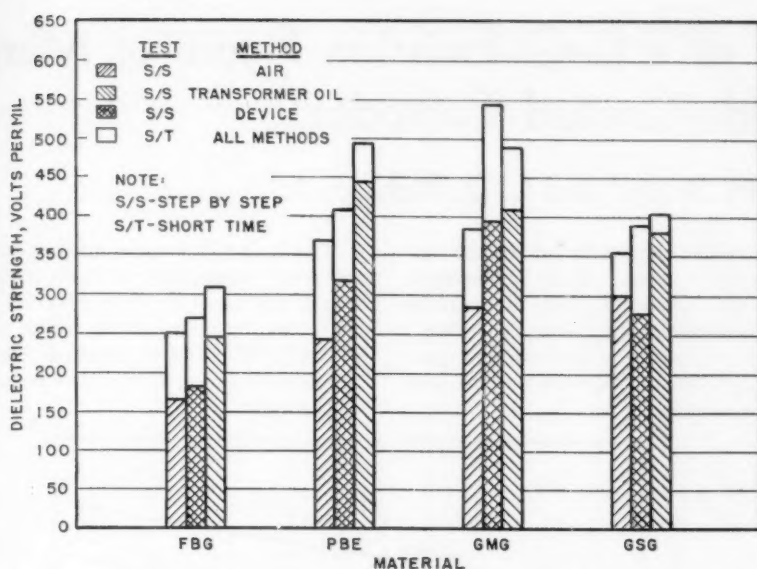


Fig. 3.—Comparison of Dielectric Strength Measured by Three Methods at 25 C., 50 per cent Relative Humidity. Values are average of test specimens.

The equipment was tested by applying a test voltage of 85 kv. to the electrodes with a high dielectric strength specimen inserted. This voltage was impressed for a period of one minute and repeated several times. The only adverse effect observed from this test was some slight corona burns on the silicone rubber gaskets in the immediate vicinity of the electrodes. When operating in the upper temperature range, it is advisable to dust the specimens with a light coating of talcum powder to prevent adhesion of the specimen to the silicone rubber gaskets.

COMPARISON TESTS

In order to determine how the results obtained with this dielectric measuring device (to be referred to as the *gasket-pressure device*) compares with results obtained by methods already known and accepted, the following measurements were conducted:

Specimens were prepared from 36 by 42 by $\frac{1}{8}$ -in. sheets of the following typical laminated thermosetting materials:

Navy Designation	Description
FBG	Fabric base, phenolic resin
PBE	Paper base, phenolic resin
GMG	Glass base, melamine resin
GSG	Glass base, silicone resin

The sheets of materials were first cut into 12 by 16 by $\frac{1}{8}$ -in. specimens for dielectric strength measurements conducted in air at laboratory ambient temperature of 25 C. and at 50 per cent relative humidity. In this, and in all

other methods of measurement, the electrodes used were $\frac{3}{4}$ -in. brass disks with edges rounded to $\frac{1}{8}$ -in. radius. Two specimens of each material were measured by the "short-time" method,³ and three specimens by the "step-by-step" method.³ These specimens were then cut into 4 by 4-in. specimens, discarding the puncture produced in the air breakdown measurements and its surrounding area. The resulting specimens were randomized, and measurements similar to the air dielectric strength measurements were conducted in transformer oil and in the gasket-pressure device. The results of these measurements are shown in bar-graph form in Fig. 3. From these data, it may be seen that for all measurements except the short-time measurement of GMG

material, the gasket-pressure device produced results considerably closer to true air dielectric strength measurements than measurements made with the test specimens in transformer oil.

Measurements similar to those conducted at room temperature were conducted at 100 C. on specimens taken from the material used in the air dielectric strength measurements. These measurements were conducted in transformer oil and in the gasket-pressure device. Determinations of true air dielectric strength were not conducted at 100 C. due to lack of facilities.

These specimens were heated for 24 hr. at 100 C. before dielectric strength measurements were conducted at this temperature. The results of these measurements are shown in Fig. 4. It may be seen from Fig. 4 that for all measurements the gasket-pressure device produced lower values of dielectric strength than those obtained in transformer oil.

CONCLUSIONS

The measurements conducted indicate that the gasket-pressure device produces dielectric strength measurements that generally are lower than equivalent transformer oil measurements and lie between measurements made in air and in transformer oil.

The gasket-pressure device has the advantage of being capable of measuring dielectric strength of small-size specimens at elevated temperatures up to 250 C. in an efficient, safe, and rapid manner.

Acknowledgment:

The authors are indebted to P. Valentine of the Material Laboratory for his skillful assistance in constructing the equipment described herein.

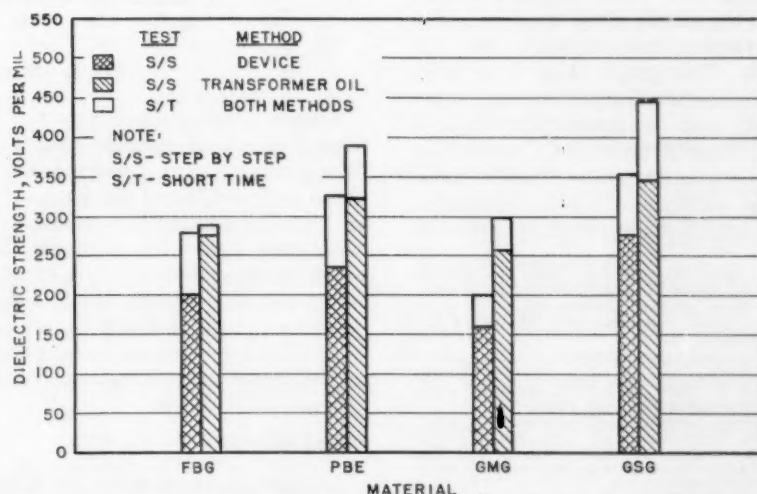


Fig. 4.—Comparison of Dielectric Strength Measured by Two Methods at 100 C. Values are average of test specimens.

Development and Use of a D-C. Emission Figure of Merit for Oxide-Coated Cathodes¹

By Thomas H. Briggs² and C. D. Richard, Jr.³

NICKEL-ALLOY cathode sleeves, when coated with barium and strontium oxides and heated to about 800 C., form the heart of most electron tubes in use today. It has long been known that variations in quality of the sleeves create vast dissimilarities in the behavior of tubes. Due to the large number of process variables and the difficulty of measuring them, the causes have never been adequately determined.

In 1945 a section of Subcommittee VIII of ASTM Committee B-4 on Electrical Heating, Resistance and Related Alloys was organized to study nickel-alloy cathode sleeves and to develop methods for measuring the performance qualities which would be reliable, simple, and rapid. Considerable progress has resulted and the studies are continuing (1, 6).⁴

As one phase of the investigations, a standard diode was adopted (2). This electron tube was stripped of all but essential parts and was produced under the closest possible controls. (See Proposed Method of Test for Relative Thermionic Emissive Properties of Materials Used in Electron Tubes appended to 1950 annual report of Committee B-4.)⁵

A graph of d-c. emission (I_s in milliamperes of current) as a function of cathode temperature (heater volts, E_f with plate voltage held constant) is used in the test work to determine a quality of the oxide-coated cathodes. Due to the simple diode structure, only the emission characteristics may be studied. This graph (Fig. 1) consists of a temperature-limited section (nearly vertical) and a space-charge-limited section (nearly horizontal), joined by a knee whose sharpness depends upon cathode quality and tube processing.

The ASTM diode investigations were

conducted by eight different laboratories over a 5-yr. period. Early in the work it became apparent that comparison of entire emission curves was necessary, since the location of the straight portions themselves was inadequate and often misleading.

The knee of the curve may be mathematically considered as the point at which the second derivative has its maximum negative value. The figure of merit (FM) is obtained through a graphical method for the determination of this point.

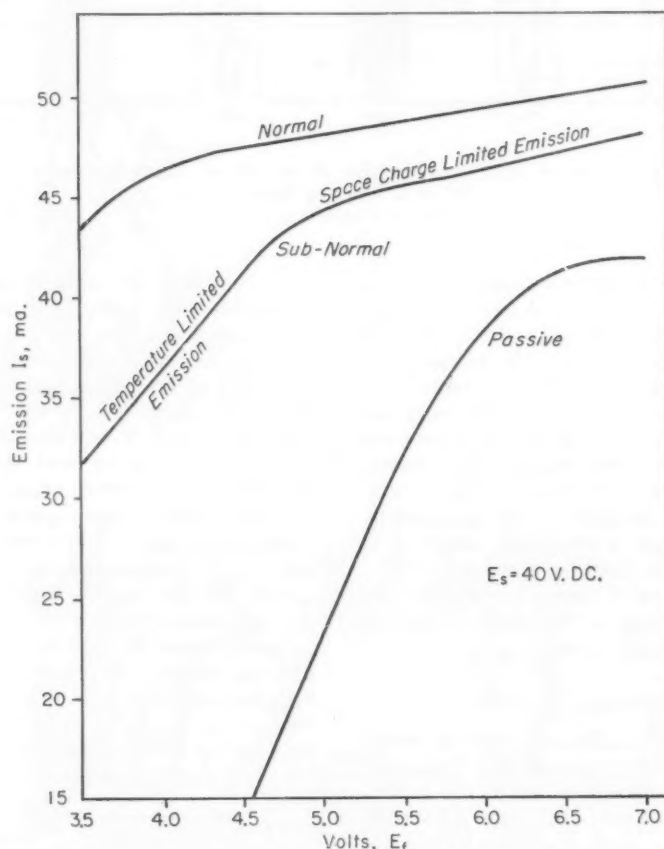


Fig. 1.—Standard Diode d-c. Emission.

Further study showed that the best over-all index of cathode quality and tube processing was connected with the shape and location of the knee. (This is supported later in this article.) The need then arose for a single figure which would express the quality of the curve, and, specifically, the location of the knee. Such a figure would make it possible to attempt correlations of several types and comparison of results between laboratories.

Figure of Merit:

A single figure of merit for the d-c. emission quality of a cathode has been developed and is now included in the diode test method proposed to the Society.

As the first step in the calculation, the E_f - I_s curve is plotted on log-log graph paper to emphasize the knee (Fig. 2). A point (1 v.- E_f , 10 ma.- I_s) is chosen as an arbitrary origin. From this point a tangent is drawn to the emission curve. The FM is the slope of this tangent line (I_s/E_f).

In running emission tests, a control lot is always included. Then it becomes desirable to state how much better or worse is the test than the control. This is done by dividing the FM for the test lot by the FM for the control lot, multiplying this quotient by ten to provide a two-digit number, and rounding off to one decimal place. This is called the quality ratio (QR).

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¹ This paper is based upon two papers originally presented before the American Physical Society on March 1, 1950. New and additional data are incorporated in this joint paper.

² Chairman, Section A of Subcommittee VIII of ASTM Committee B-4; Research Division, Burroughs Adding Machine Co., Philadelphia, Pa.; formerly Electronics Laboratory, Superior Tube Co., Norristown, Pa.

³ Member B-4-VIII-A, Electronics Laboratory, Superior Tube Co., Norristown, Pa.

⁴ The boldface numbers in parentheses refer to the list of references appended to this paper.

⁵ Report of Committee B-4 on Electrical Heating, Resistance and Related Alloys, *Proceedings, Am. Soc. Testing Mats*, Vol. 50, (1950).

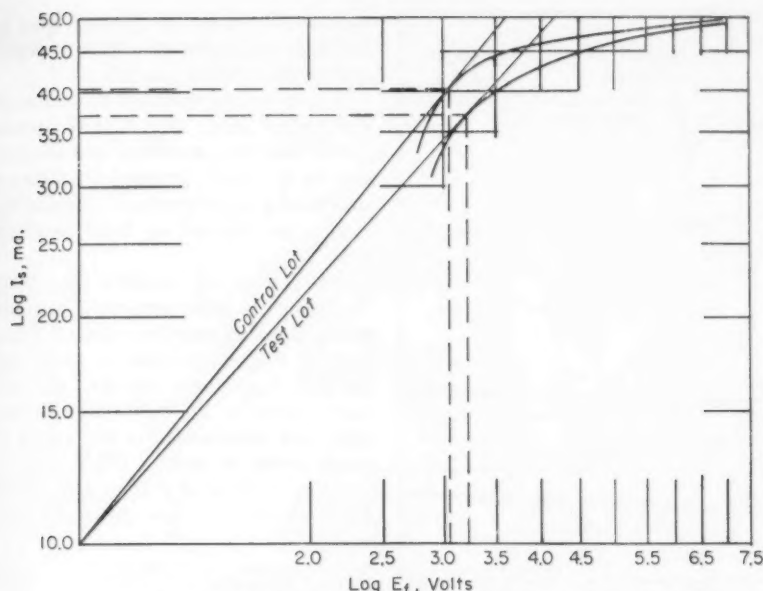


Fig. 2.—Log-Log Presentation of I_s versus E_f ($E_s = 40$ v. d-c.).

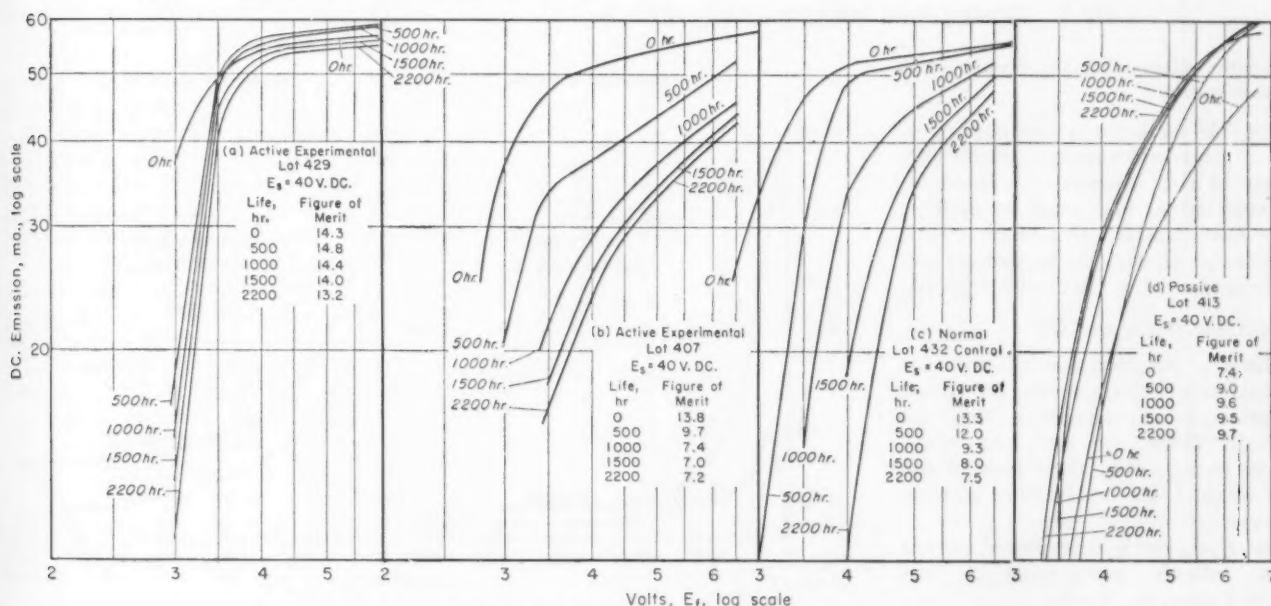


Fig. 3.—Log E_f versus Log I_s versus Life.

As an example, take the data from Fig. 2:

Test lot coordinates of tangent point:

$$E_f = 3.2 \quad I_s = 37.0 \quad FM = \frac{37}{3.2} = 11.6$$

Control lot coordinates of tangent point:

$$E_f = 3.03 \quad I_s = 40.3 \quad FM = \frac{40.3}{3.03} = 13.3$$

$$\text{Quality Ratio} = \frac{\text{Test FM}}{\text{Control FM}} = \frac{11.6}{13.3} \times 10 = 8.7$$

(Test is poorer than control.)

It must be understood that the FM values of control lots for different series of tests will vary considerably. This has been shown to be due to variables which have not been capable of control, so far. A control lot is judged on the

basis of the shape of the E_f - I_s curve, the spread between its individual tubes, and the saturation I_s value at rated cathode temperature. These criteria are compared with previous experience.

Then the FM values provide single figures for evaluation of the test lots with the direct control. Thus, the unavoidable processing variables are neutralized to the greatest possible degree. The QR values are useful in comparing different series of tests, each one having its own control lot.

By comparison of the QR for different types of cathode material, such as normal and passive alloys, good agreement is obtained with ratings based upon actual production experience.

Much worthwhile information can also be obtained from a comparison of

different melts of the same alloy and different alloys of similar quality. Excellent agreement has been obtained between the QR for the diode tests and results reported in the melt approval program.

The following are some of the figures proposed in the past with a few words explaining why they were rejected (reference to Fig. 1 may aid in understanding these rejections):

(a) Emission current at a particular value of heater voltage.

1. High values of E_f are an indication of processing and will not differentiate between similar melts.

2. Low values of E_f give emission readings which are too unstable for valid comparisons.

(b) Heater voltage for a particular value of emission current.

1. Too difficult to read because of time lag between voltage setting and temperature stabilization.

2. The objections raised against (a) are true here also.

(c) Tube drop for a particular value of emission current.

1. Too unstable if taken at low current.

2. Of little value if taken at high I_s since that is in space charge-limited region.

(d) Drop in emission current per volt change in heater voltage over a specified E_f range. Does not show correct differences between good and very poor lots.

(e) Heater voltage change required to give a definite or a percentage drop in emission current—same reasons as for (d).

(f) Area under curves.

1. Laborious and difficult calculations.

2. Affected greatly by variations in tube geometry.

(g) Drop in emission current over a specified drop in heater voltage times the emission current for a particular value of

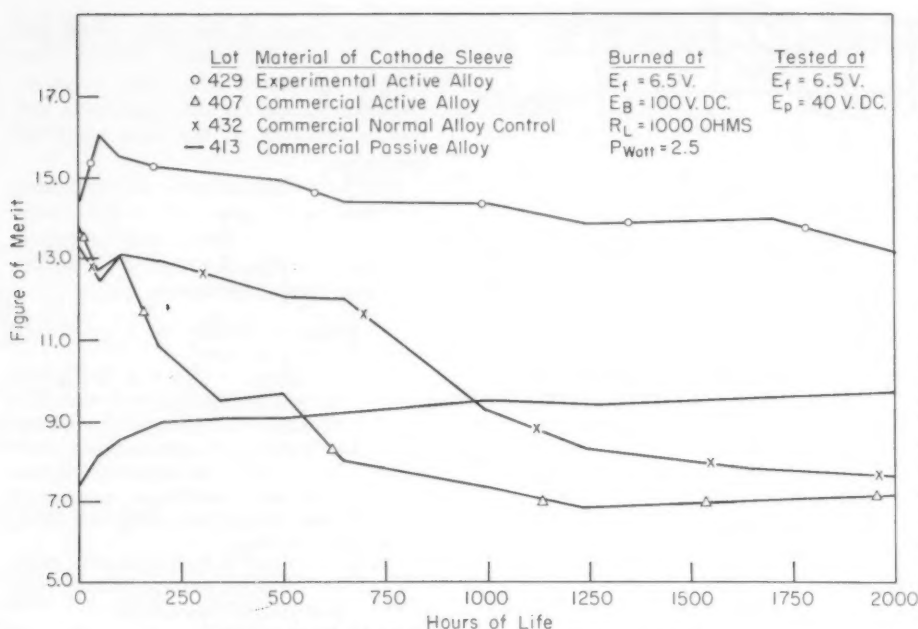


Fig. 4.—Standard Diode Life versus Figure of Merit.

the heater voltage—has not been tried as far as is known.

The *FM* method requires plotting of curves and calculations which are somewhat time consuming. However, the correlations and range of applications have justified this effort. It is intended as an engineering method and is obviously not scientifically rigorous.

Application of *FM* and *QR*:

Through consideration of the d-c. emission of a cathode, several important performance characteristics may be studied. These are discussed below and serve to justify the choice of the *FM* method for evaluating cathode behavior.

The E_f - I_s curve knee moves toward lower cathode temperature as the quality improves. During life it moves toward higher temperatures and generally loses much of its sharpness.

Figure 3 shows typical graphs of four grades of cathode alloys and the calculated *FM* values. Despite the closeness of the curves in Fig. 3(a), the *FM*'s calculate to significantly different values. The curves of Fig. 3(b) and (c) show steady movement during life to higher temperature regions and with progressively broader knees. The curve in Fig. 3(d), for a passive alloy, moves to lower temperature regions with increasing life. Note that the knee remains broad. This indicates a narrow permissible range of operation in the space charge limited region. On the contrary, Fig. 3(a) shows a wide operating range.

Figure 4 shows the *FM* values for the same four alloys plotted against time. The excellent spread of d-c.

as shown by the *FM* method, may be a function of inadequate tube processing rather than sleeve quality.

When several melts of the same alloy are tested using the ASTM standard diode, their d-c. emission and life curves can be so closely grouped that they are practically superimposed. Again, these results are backed by tube production data.

One word of caution is needed. Variations in tube geometry, materials, and processing must be controlled to the highest degree to permit truly comparable test diode results (6). It is even desirable to run many tests at once and compare only the tubes of a single series of tests. With these precautions, values of *FM* or *QR* differing by more than 0.5 are probably significant.

Rate of Activation:

The rate of cathode activation is of particular interest in the use of passive

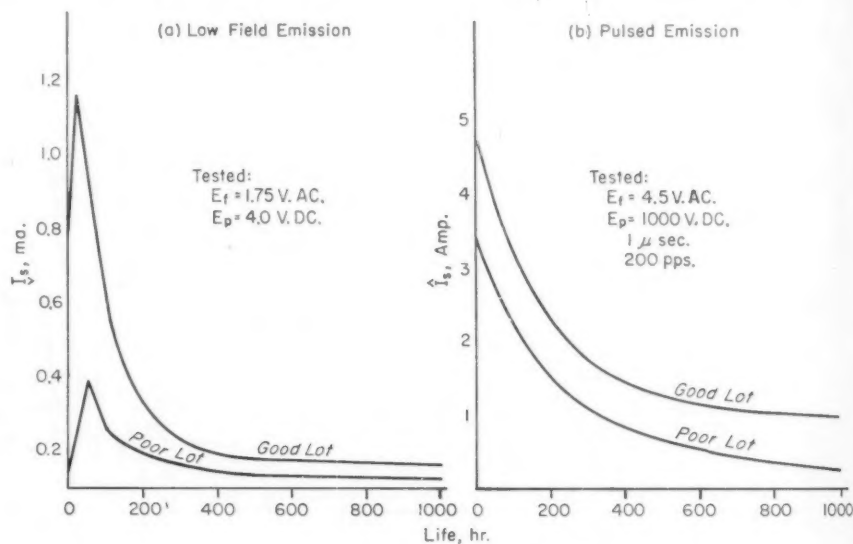


Fig. 5.—Standard Diode Life Test.

emission as indicated by the *FM* results is clearly demonstrated. These relative effects have been borne out in actual production tubes when rated on the basis of G_m or similar tube characteristics.

The simplicity of indicating life performance through *FM* curves is also shown. Little change would be denoted by the saturation values (Fig. 3), and that lack of change would not be reflected by production experience. Even a plot of some substandard cathode temperature (that is, $E_f = 4.5$ v.) would not be truly indicative, since sometimes it is on the knee and at other times is either temperature- or space-charge limited.

The droop of the control line, and also of the other lots, at late hours of life,

alloys and in studying the effects of migratory reducing agents, or the relative reducing powers of various elements in the base metal. The most sensitive measurement of change, and the one most closely correlated with standard radio receiving circuit application, is the d-c. emission quality as determined by movement of the knee of the E_f - I_s curve.

For standard processing, cathodes of different sleeve compositions will activate to different degrees. Standard life test burning will eventually achieve the optimum of activation, following which there is generally a long time of stable emission. The time on life to reach the optimum emission is a relative measure of the rate of activation.

Figure 4 shows that two commercial

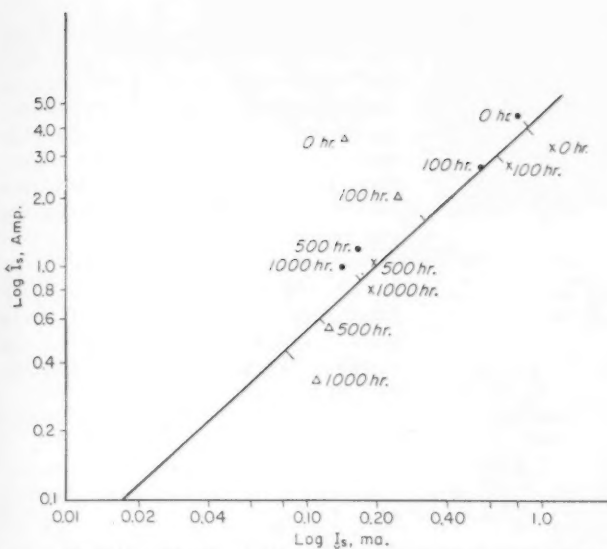


Fig. 6.—Standard Diode I_s versus I_a Correlation Changes with Life.

alloys were at an optimum following the schedule used. The experimental active alloy started higher but required 50 hr. to come to full activity. The passive alloy only reached stability after 250 hr. of life.

If the processing schedules were altered, the speed of activation could be made to be the same. So this is a relative measure only, and a way to indicate schedule requirements.

Cardell (4), has reported that passive cathodes activate more rapidly and to a higher level if the anode is of Grade "A" nickel. If a similar passive nickel is used for the plate, then the passive cathode activates slowly and slightly. The curves of Figs. 3(d) and 4 (lot 413) are for passive cathodes with passive plates. When previously run with Grade "A" nickel plates, the same cathodes activated almost to the initial level of the "normal" cathode alloy control lot. This, we believe, is excellent confirmation of Cardell's results using "low field" and pulsed emission measurements.

Pulsed and Low Field Emission:

In our work the greatest emphasis has been placed upon d-c. emission at $E_p = 40$ v. It offers the greatest uniformity of values between tubes in the same lot. Changes during life are more gradual and uniform. Receiving tube cathodes must generally meet this condition.

Pulsed emission is important for certain applications; and as is well known, the cathode alloy most favorable for pulsed applications is not necessarily the best for d-c. drain.

Low field emission testing was developed by Nottingham, McCormack, and Cardell (3). Its purpose is to obtain stable d-c. emission conditions at very low cathode temperatures

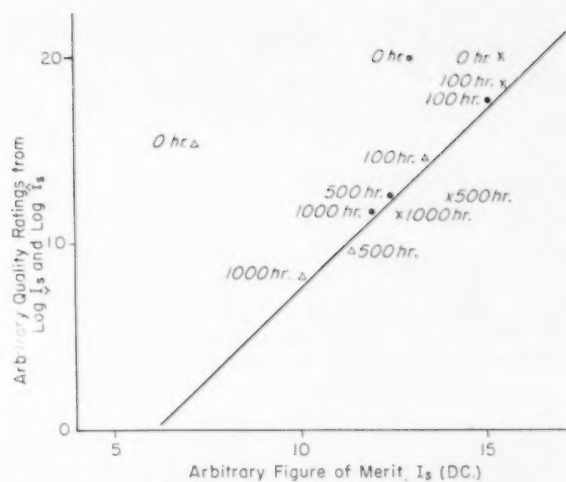


Fig. 7.—Standard Diode Correlation of Quality Ratings from Data on I_s , I_a , I_s and Changes with Life.

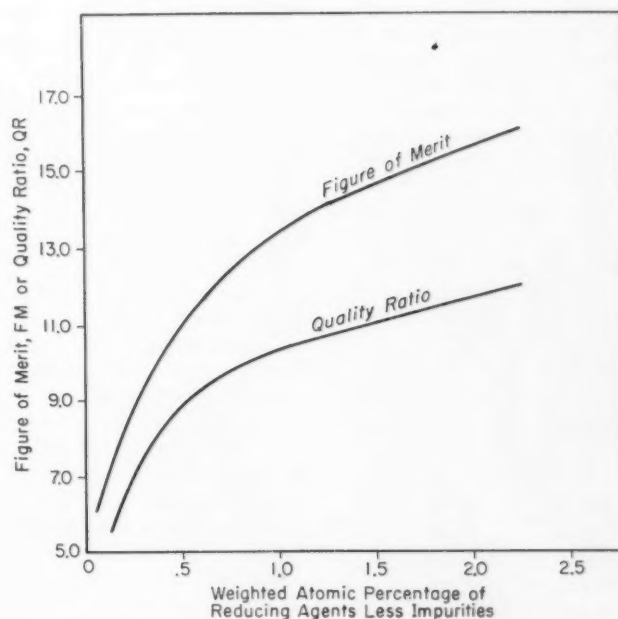


Fig. 8.—Diode d-c. Emission Figure of Merit of Quality Ratio versus Weighted Atomic per cent Composition of Cathode Sleeve.

(about 375 C.). This requires the use of an anode potential low enough so that the energy of the electron stream will not decompose the oxide layers deposited upon the anode surfaces during activation. In other words, it aims to eliminate this source of cathode poisoning.

Figure 5 shows life performance of a good and a poor lot of tubes for both pulsed and low field emissions. Life burning is under d-c. emission test conditions.

The hyperbolic shape of both curves is indicative of early hour transient effects. The range of values within any given test lot varies tremendously. This is not peculiar to diodes produced only in our own laboratory.

In our opinion all three types of emission test must be considered in parallel when judging cathode performance and quality. We believe that each one is indicative of primary action by a different portion of the cathode assembly.

Figure 6 shows a log-log plot of low field and pulsed emission. This correlation was first described by Nottingham, McCormack, and Cardell (3). Our data confirm their finding of the linear relationship. The figures adjacent to the plots of the lot averages indicate the life test reading period.

Since our activation schedule is lenient, there is some initial instability which adversely affects the plot grouping at 0 hr. After seasoning on the

TABLE I.—CATHODE ALLOY COMPOSITION.

Lot	Copper	Iron	Manganese	Carbon	Magnesium	Silicon	Titanium	Other
PER CENT BY WEIGHT								
413—Passive.....	0.013	0.018	0.002	0.010	0.004	0.009	0.001	0.002
432—Normal.....	0.07	0.06	0.12	0.002	0.04	0.04	0.03	0.010
407—Intermediate.....	0.015	0.078	0.056	0.038	0.009	0.182	0.004	0.006
429—Experimental active.....	0.011	0.019	0.002	0.019	0.043	0.035	0.003	4.15*
ATOMIC PER CENT								
413.....	0.012	0.019	0.002	0.049	0.010	0.019	0.001	0.004
432.....	0.065	0.063	0.128	0.05	0.097	0.084	0.037	0.022
407.....	0.014	0.082	0.060	0.186	0.022	0.382	0.005	0.013
429.....	0.010	0.020	0.002	0.095	0.104	0.074	0.004	1.40*
WEIGHTED ATOMIC PER CENT								
	Impurity Total Cu-Fe-2 x Mn	1 x C	5 x Mg	3 x Si	1 x Ti	1 x Other	Reduce Total	"Reducing Potential" (Reduce Total less Impurity Total)
413.....	0.035	0.049	0.050	0.057	0.001	0.004	0.161	0.136
432.....	0.384	0.050	0.485	0.262	0.037	0.022	0.856	0.472
407.....	0.216	0.186	0.110	1.146	0.005	0.013	1.460	1.244
429.....	0.034	0.095	0.520	0.222	0.004	1.400	2.241	2.207

* Purposeful additive is Tungsten.

life rack, adherence to the slope line is markedly improved.

Possibly the high initial \bar{I}_s for Δ lot is due to abnormally low interface resistance, which does develop as activation reaches the optimum during life.

As life test hours increase, cathode activity drops and the plot moves closer to the origin. Thus, relative location of various lots measured along this slope line may be taken as an arbitrary measure of relative quality for pulsed and low field emission.

Correlation of Emission Qualities:

It has generally been considered that there is no relationship between pulsed and d-c. emissions. Through the use of our figure of merit for d-c. emission quality we believe we have shown that there is a correlation.

From Fig. 6 we obtained arbitrary quality ratings for \bar{I}_s versus J_s by measuring, from an origin, the location on the slope line of the projections of the various lots.

This origin is the point of intersection of the slope line with the abscissa of Fig. 6. The ordinate of Fig. 7 is then the projected location of the test points measured along the slope line of Fig. 6.

These data were plotted against the FM for d-c. emission and are shown in Fig. 7. Here again, initial instability is reflected by widespread location of zero hour readings. Between 100 and 1000 hr., the grouping along a straight line is excellent.

Possibly this can have real significance in evaluations of test lots and study of

their relation to theory of thermionic emission.

Alloy Composition versus Emission:

An important phase of cathode research is to obtain an understanding of the role of the base metal composition as it affects thermionic emission.

From many tests and from production experience it is known that certain elements are several times more active in reducing the oxide coating than are others. Further, as the level of alloy impurities increases, the reducing agent content must be increased to provide the same d-c. emission level.

It is impossible as yet to assign accurate weighting factors for the reducing ability of each element. It will be necessary, first, to determine the ratio of compounded to uncompounded reducing agents in the original cathode sleeves.

Working in reverse, we can estimate weighting factors on the basis of experience and apply these to known alloys run under comparable conditions. These weighting factors will be a function of reducing power of each element, the ability to diffuse to the surface of the metal sleeve, and the amount present in active form. If these factors provide data which correlate with test emission, then the problem of working backward to evaluate the parameters is simplified.

A very rough first approximation of a "reducing potential" for four grades of alloys is shown in Table I and Fig. 8. The computation steps are shown in the table. The graphs are for the reducing potentials (RP) versus both FM and

QR at 0 hr. Other weighting factors are less successful. Later hour emission values cannot be used due to migration alterations of alloy composition—particularly with respect to magnesium and to additives from external sources. This method has also been used successfully with many other cathode lots and alloys.

No claim is made to scientific accuracy, but it is an indication of how the d-c. emission figure of merit can be used in connection with the reducing potential to evaluate the emission characteristics of cathode alloys.

Conclusion:

The complex problem of cathode base metal behavior has been separated into several simpler problems, through cooperative effort by members of Committee B-4. The use of an arbitrary d-c. emission figure of merit (FM) permits evaluation of cathode performance by a simple number. Using this numerical rating, it is possible to obtain clear differentiation of life testing, rate of cathode activation, and correlation of d-c. emission with pulsed and low field emission. Most important, the FM method has led to a first rough approximation of a cathode alloy's reducing potential (RP). When this is known with some certainty, it will be possible to predict the effect of a nickel alloy in an oxide-coated cathode without resort to the present lengthy, expensive, and unreliable tube testing methods.

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Application of X-ray Diffraction Methods to the Identification of Natural and Synthetic Rubbers¹

By S. Goldspiel² and F. Bernstein²

SYNOPSIS

In this paper, X-ray diffraction patterns and data for rubber compounding ingredients are presented and applied to the identification of major crystalline ingredients in several basic rubber stock types. Methods for the identification of natural and synthetic elastomers by low-temperature aging or stretching using film techniques are described. The extension of such methods to the Geiger counter X-ray spectrometer is presented and discussed.

SCOPE

This paper discusses the development of applications of X-ray diffraction methods to the identification and study of natural and synthetic rubber stock types as part of a broad elastomer research program. The investigations conducted to date include the following applications:

1. Film techniques for the identification of solid compounding ingredients for subsequent use in qualitative analysis of rubber stock patterns.
2. Film techniques for qualitative identification of representative rubber types.
3. Film techniques for the quantitative estimation of crude and reclaimed rubber mixtures.
4. Geiger counter spectrometer techniques for the qualitative identification of representative rubber types.

THEORY

The theory of X-ray diffraction is based on the fact that when X-rays of a particular wave length strike a substance, a scattering of the radiation results so that it appears at every possible angle to the incident beam. If the scattering substance is a crystal, the scattered radiation is much more intense at certain angles than at others; that is, the X-rays are diffracted through definite angles. The angles of diffraction, θ , depend upon the wave length of the X-rays used and the arrangement and spacing, d , of the atoms of the crystal. W. H. and W. L. Bragg in their classical work on X-rays and crystal structure found that a crystal acts toward X-rays like a family of

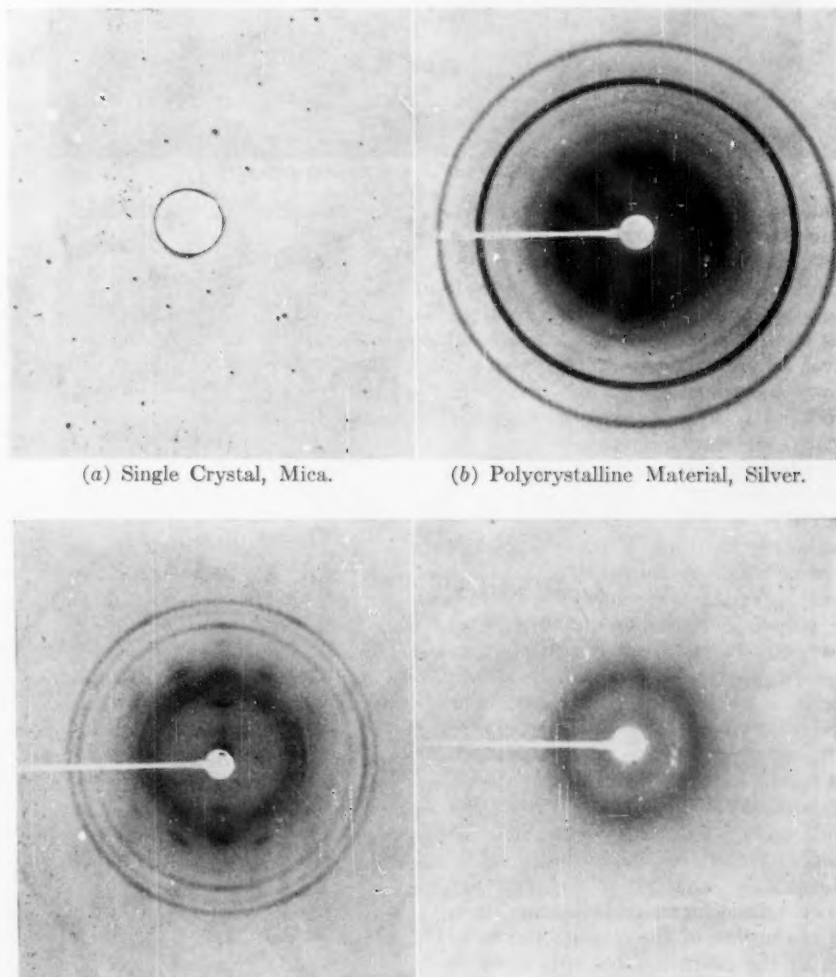
plane gratings placed one above the other. They derived the equation known as Bragg's law, namely,

$$n\lambda = 2d \sin \theta$$

where n is the order of diffraction, λ is the wave length of the X-rays, and the other symbols have the significance indicated previously. The methods of

recording the diffraction patterns, of measuring the parameters used to determine the diffraction angles, θ , for the computation of spacing between atomic planes, d , producing the diffraction maxima, as well as of the relative intensities of these maxima, form the bases of various studies in X-ray diffraction.

The appearance of a diffraction pattern depends upon such factors as crystallinity, particle size, and orientation of the material irradiated. Figure 1 shows four general pattern types. A single crystal such as mica (upper left) produces a pattern of sharp spots, the locations of which are defined by the particular planes which are correctly



(a) Single Crystal, Mica. (b) Polycrystalline Material, Silver. (c) Fibrous Material, Stretched Rubber. (d) Pseudo-amorphous Material, Polyvinyl Formal.

Fig. 1.—Quality of X-ray Diffraction Patterns.

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¹The opinions expressed herein are those of the authors and do not necessarily represent those of the Navy Department.

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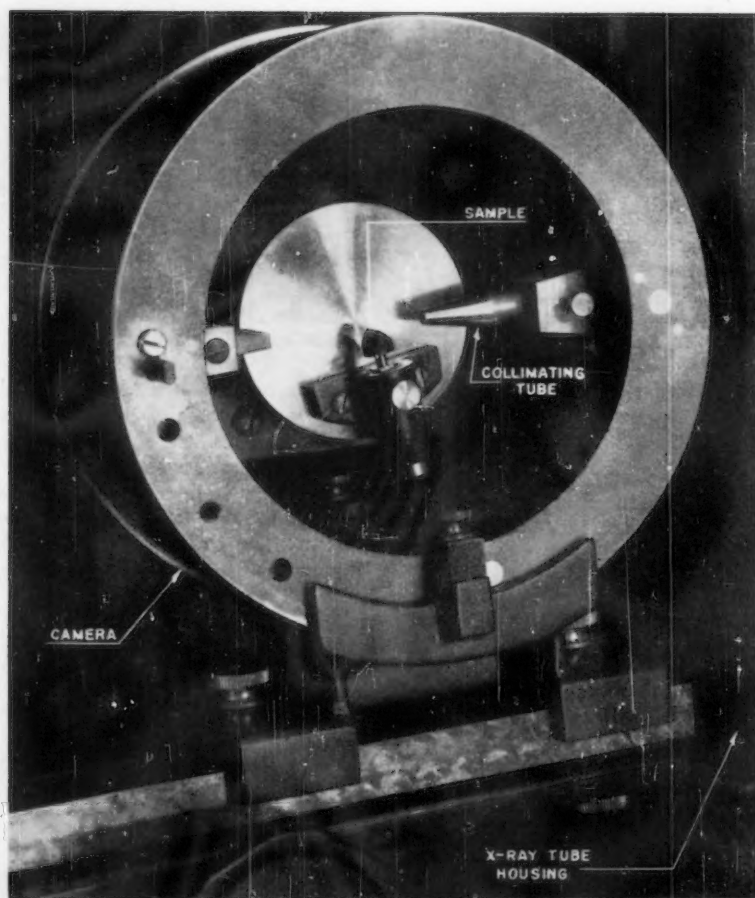


Fig. 2.—Test Setup for Compounding Ingredients.

situated with respect to the primary beam to satisfy the Bragg angle. A powder sample in which there is a random distribution of planes results in the merging of spots to form a continuous circle, as exemplified by powdered silver (upper right). If the material shows directional orientation, the effect produced is a series of symmetrically located crystal interference spots as indicated by the pattern for stretched rubber (lower left). Pseudo-amorphous materials produce diffuse halos or bands, the location of which is related to some fundamental periodicity in the structure of the material. The pattern for polyvinyl formal is an example of this group of materials, which includes uncrystallized rubber as will be shown later. In general, then, for a particular type of material, the quality of the diffraction pattern produced is dependent on the inherent order in the atomic arrangement or the degree of orientation produced by external effects such as temperature, stress, or work.

Qualitative analysis of materials by X-ray diffraction methods depends upon the evaluation of the quality and location of the diffracted beam images, as recorded by photographic film or by direct reading instrument. The location of the diffraction images is uniquely a function of the material and conse-

quently may be utilized in the identification of it. The pattern of a mixture, furthermore, is a simple addition of the patterns of the individual contributing components. Quantitative analysis of materials by X-ray diffraction is based on the fact that the intensity of a particular diffraction image is a function of the amount of material producing it.

COMPOUNDING INGREDIENT IDENTIFICATION BY FILM TECHNIQUES

X-ray diffraction methods have been used in various studies of rubber compounding ingredients (1, 2).³

A comparison of a list of the various compounding ingredients for the several stocks furnished for this investigation, with the ASTM Index of Diffraction Patterns (3), showed, however, that data for only a few of the compounding ingredients have been catalogued. It was therefore necessary to prepare and index patterns, where such were unavailable, for all the compounding ingredients in the uncombined form.

The powder method of X-ray diffraction analysis was used in these studies. It was devised independently by Debye and Scherrer (5) and Hull (6), and provides a means for rapid and positive identification of crystalline substances. The method consists of mounting a sample of material at the

³ The boldface numbers in parentheses refer to the list of references appended to this paper.

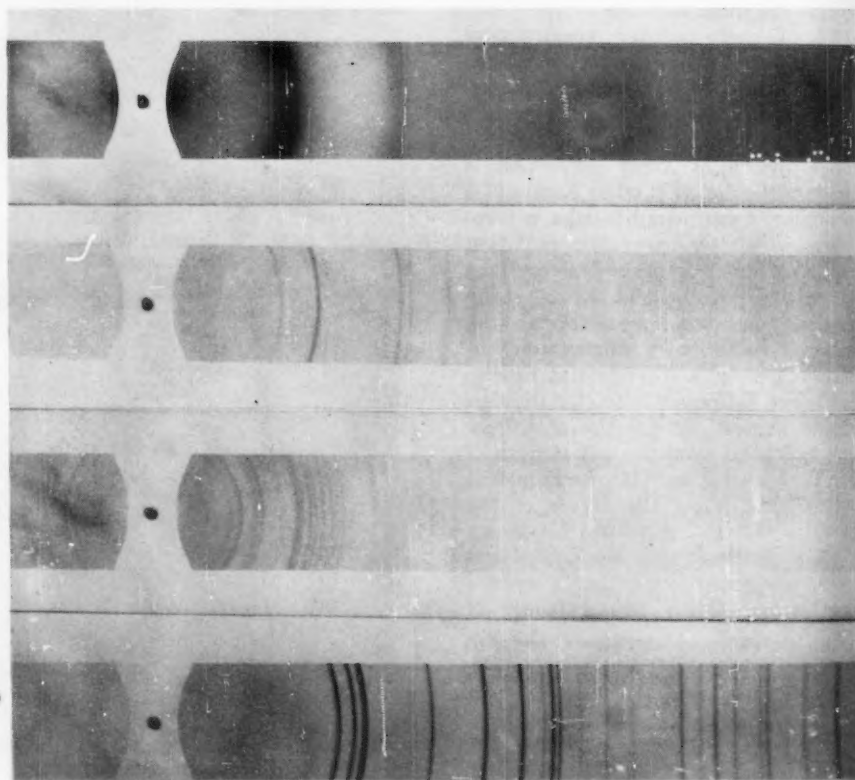


Fig. 3.—Representative X-ray Diffraction Patterns for Compounding Ingredients.
(a) Thermax; (b) Vandex; (c) Thionex; (d) Zinc Oxide

TABLE I.—X-RAY DIFFRACTION DATA FOR COMPOUNDING INGREDIENTS.

Material	<i>d</i>	<i>I</i> / <i>I</i> ₀	Material	<i>d</i>	<i>I</i> / <i>I</i> ₀
Altax.....	13.5	1.00	Neozone A.....	4.40	1.00
	5.45	0.73		4.51	0.96
	3.56	0.72		3.60	0.96
	3.99	0.41		11.4	0.83
	3.08	0.33		3.50	0.67
Captax.....	7.60	1.00	Polyac.....	3.54	1.00
	3.13	0.87		7.12	0.97
	3.76	0.78		5.63	0.83
	3.19	0.67		4.43	0.66
	3.84	0.49		2.34	0.55
Heliozone.....	4.13	1.00	Stearic acid....	4.13	1.00
	3.72	0.71		3.75	0.10
	2.48	0.13		3.99	0.05
	4.58	0.09		4.35	0.04
	2.22	0.09		13.2	0.03
Diphenyl-Guanidine...	4.63	1.00	Tuads.....	3.42	1.00
	10.3	0.58		5.62	0.94
	4.14	0.32		4.95	0.37
	3.47	0.31		4.73	0.36
	4.46	0.28		6.27	0.21
Litharge.....	See A.S.T.M. Data Card No. 1999(3) or 1981(4)		Thionex.....	5.85	1.00
Magnesia.....	See A.S.T.M. Data Card No. 3286(3) or 3424(4)			3.58	0.72
Sulfur.....	See A.S.T.M. Data Card No. 1131(3) or 1082(4)			6.54	0.49
Vandex.....	See A.S.T.M. Data Card No. 2070(3) or 2118(4)			3.45	0.49
Zinc Oxide.....	See A.S.T.M. Data Card No. 2911(3) or 3079(4)			4.69	0.48

center of a cylindrical camera which contains photographic film. Monochromatic X-radiation is directed at the sample. The diffracted rays leaving the specimen generate a series of cones concentric with the primary beam, each of which has a semiapex angle equal to twice the Bragg angle of diffraction, θ . These diffracted rays produce a series of rings concentric with the central spot, from the diameter of which the corresponding Bragg angles and *d* values of the reflecting planes producing them can be computed, since the wave length of the radiation is known. To complete the identification data, relative line intensity values are determined from measurements made by the use of a suitable densitometer.

The compounding ingredient samples were first ground to approximately 200-mesh powder size and introduced into a wedge-shaped holder which was accurately centered in the camera. The setup is shown in Fig. 2. The length of exposure was 2 hr. in each case using screenless X-ray film and copper K α radiation at 35 kv.p. and 20 ma. The sample was oscillated through 20 deg. at constant angular speed. Sample patterns of some of the compounding ingredients are shown in Fig. 3, and the X-ray diffraction data for all those studied are shown in Table I.

The pattern of the rubber stocks, prepared for the determination of the compounding ingredients, were obtained by centering a small strip of rubber in the camera as shown in Fig. 4. The sample is rotated continuously during the exposure to assure randomization of reflecting planes and consequently smooth diffraction rings. A visual comparison of the rubber stocks and compound ingredient patterns was made

to determine which of the compounding ingredients could be detected in the rubber stocks. An example of such a comparison is shown in Fig. 5. The

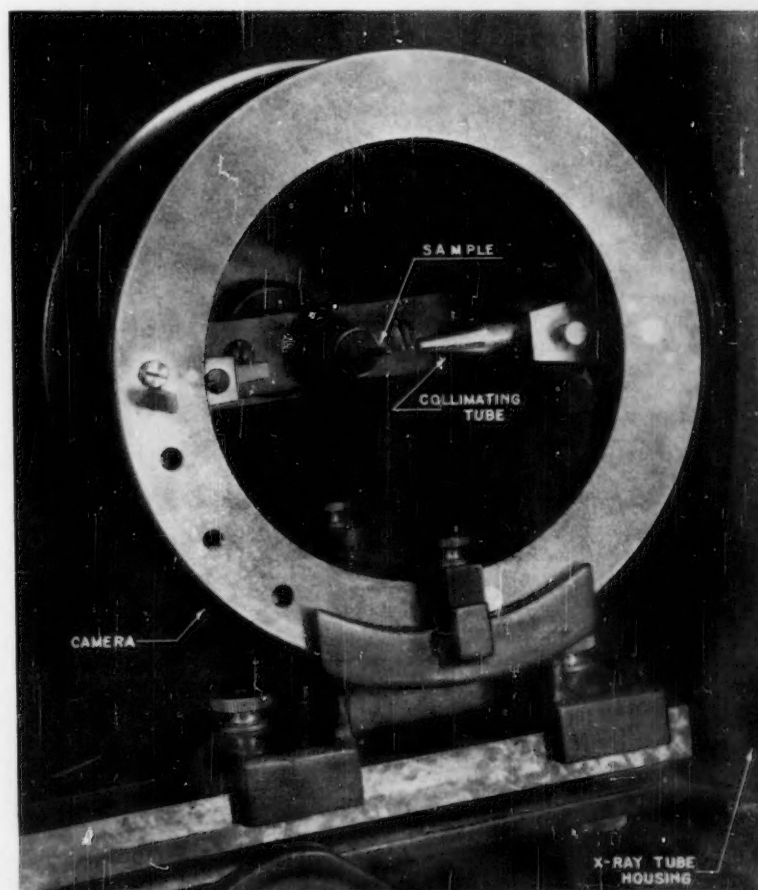


Fig. 4.—Test Setup for Compounding Ingredients in Rubber Stocks.

results show that major solid compounding ingredients which remain in the uncombined form after curing of the elastomers can readily be identified by this method. Such factors as crystallin-

ity, absorptivity, amount present, and retention of form affect ease of identification of compounding ingredients in rubber stocks. The method has the advantage over chemical methods in that it yields information about the crystalline or molecular form in which a constituent is present. It may be interesting to note that a comparison of six commercial compounding ingredients: namely, *Pelletex* (SRF), *Micro-ner*, *P-33* (FT), *Philblack A*, *Statex B*, and *Thermax* indicated that they are all nearly similar, being low-crystallinity carbon, although closer studies (7) have shown that no two carbon blacks give the same X-ray patterns.

IDENTIFICATION OF RUBBER TYPES BY FILM TECHNIQUES

Room Temperature Studies:

The fundamental criterion to be used in the appraisal of X-ray diffraction methods for the identification of natural and synthetic rubbers is whether they can be made to yield distinctive patterns. Natural and synthetic rubbers are

commonly classed as amorphous as opposed to crystalline substances. Thus an X-ray pattern of unstretched rubber is characterized by blurred rings or halos at low Bragg angles. Under these con-

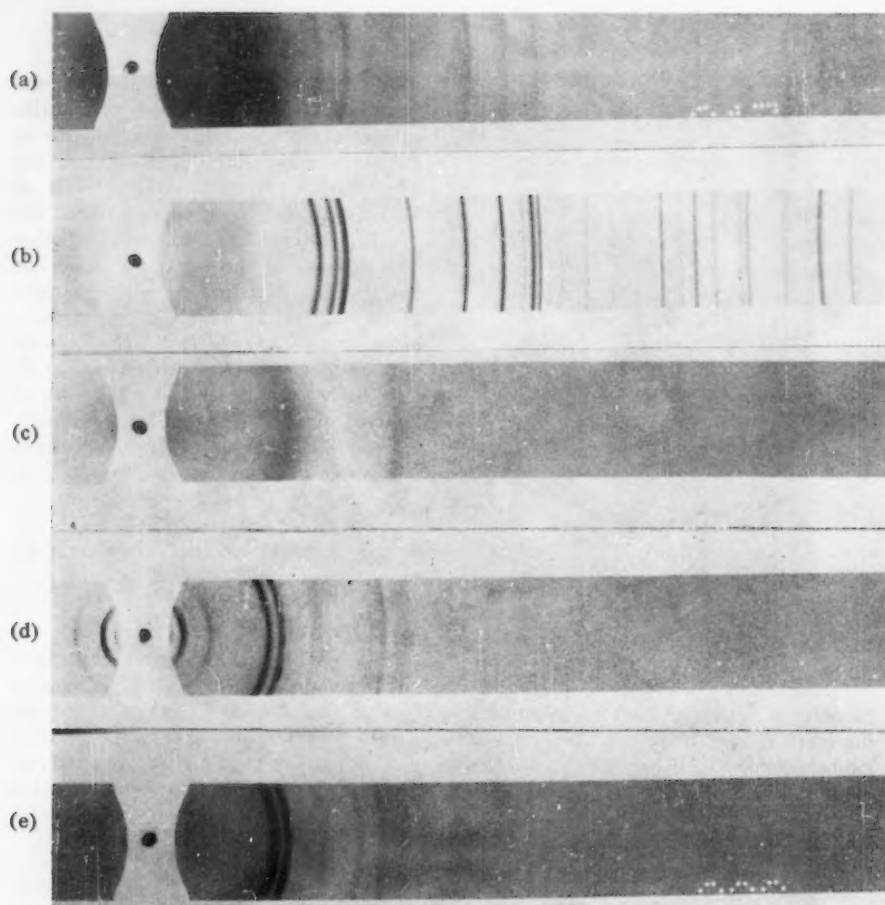


Fig. 5.—Compounding Ingredients in Nitrile Rubber.

(a) Pattern of rubber compound with Zinc Oxide, P-33 (FT), Stearic Acid, and Heliobzone (b) to (e) Patterns of commercial compounding ingredients Zinc Oxide, P-33 (FT), Stearic Acid, and Heliobzone, respectively.

ever, it has been shown in recent years that under suitable conditions of stretching, temperature, and pressure, some of these materials show properties associated with crystallinity, while others do not. Katz (8, 9) first applied X-ray diffraction methods to the study of stretched natural rubber and showed that it produces a fiber pattern with sharp interference spots. The positions of these spots are independent of the degree of stretching, but their intensities increase proportionally with it. Mark and Whitby (10) have reported that crystallization could be induced in some rubber types by subjecting them to specific low-temperature treatments. Hanson and Halverson (11) and Beu, Reynolds, Fryling, and McMurphy (12) have reported evidences of crystallinity in some rubber materials upon temperature reduction and stretching.

Even in cases where no crystallinity was observed, a high degree of stress was found to produce intensification of the amorphous halos (13, 14). It should be noted that crystallinity results referred to above were obtained by different methods including thermal, optical, mechanical, specific volume, and X-ray diffraction measurements.

In view of the above findings, it was decided to investigate the diffraction patterns for the representative rubber stocks under various combinations of stretching and low temperature aging with a view of determining the best conditions for clear identification.

Since elastomers produce X-ray reflections at low Bragg angles, all patterns were recorded on flat films using transmission techniques with the result that the entire diffraction cone is recorded. Uniform thickness samples of each of the rubber stocks were stretched to approximately 400 per cent elongation in a special sample holder as shown in Fig. 6. The patterns were taken using copper K_{α} radiation of 35 kv.p., 20 ma., screenless film, specimen to film distance of 5 cm., and an exposure time of $2\frac{1}{2}$ hr. Sample patterns for Hevea and GR-S in the stretched condition are shown in Fig. 7.

A study of the several rubber stock patterns run in the stretched condition at room temperature as indicated above shows that distinctive fiber patterns are obtained for Hevea, GR-M, and GR-I, whereas little change in the diffuse halos is noted for GR-S, Nitrile, and Thiokol.

Low-Temperature Film Studies:

In order to study the elastomers at low temperatures, a special sample holder conditions patterns for different types are not readily distinguishable. How-

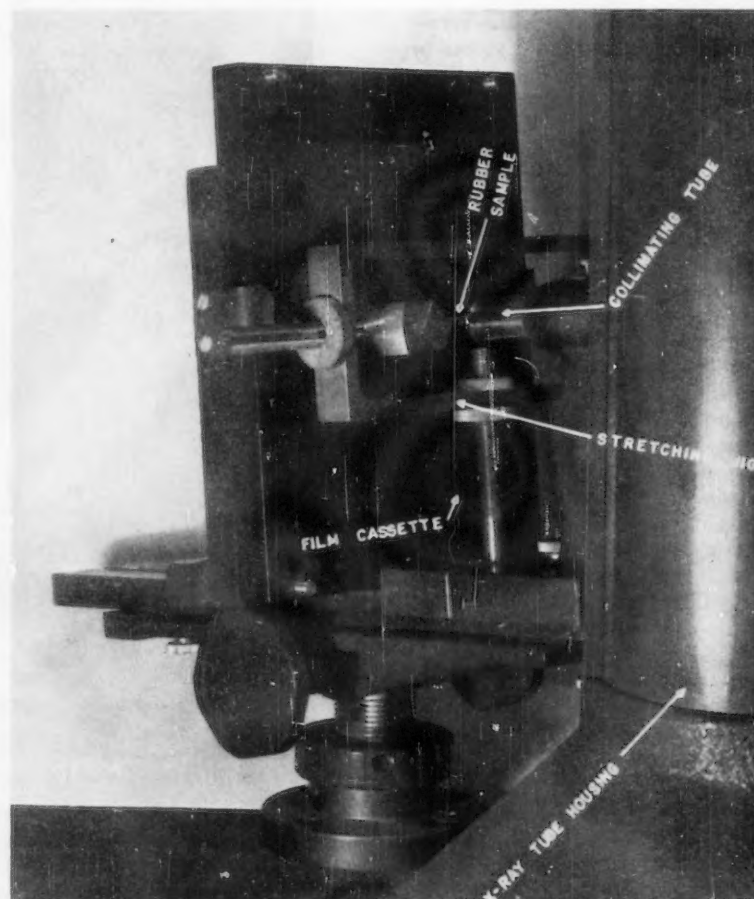
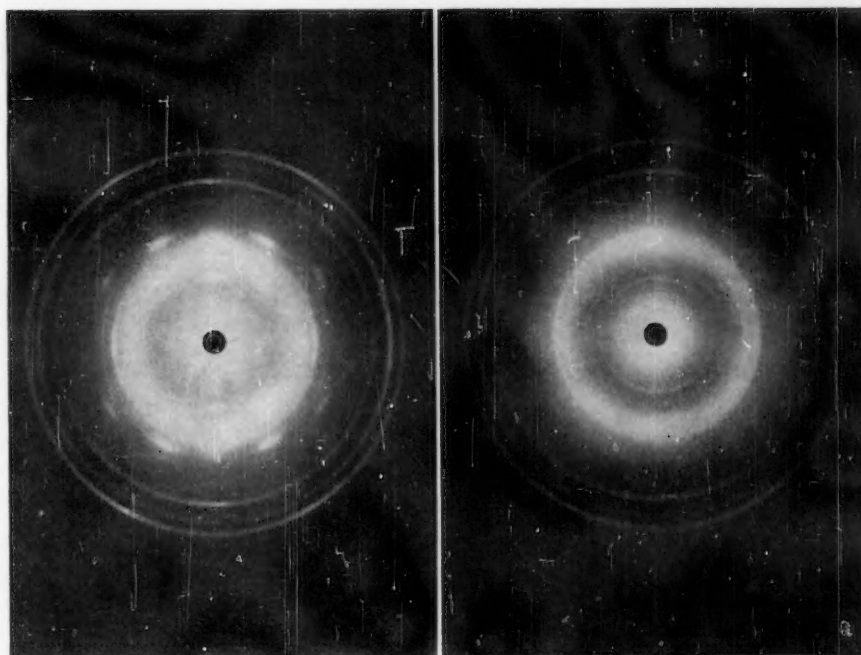


Fig. 6.—Test Setup for Elastomer-Type Identification.

with provision for cooling was designed and constructed. Photographs of the cooling unit, experimental setup, and sample holder are shown in Figs. 8, 9, and 10, respectively (6).

The operation of the cooling unit is evident from a reference to Fig. 8. The coolant which is acetone, is circulated by means of a $\frac{2}{3}$ -gal. per min. gear type pump, 2, driven by a $\frac{1}{6}$ -hp. motor, 1. The coolant is passed through coils immersed in a bath, 3, of acetone and dry ice from which it flows through a thermostat, 5, to the block, 6, and returns to the pump. When the circulating liquid reaches the desired temperature, as controlled by the setting of the thermostat, the solenoid valve, 4, in the by-pass line is energized. This causes most of the coolant to by-pass the cooling coils. The flow through the cooling and by-pass circuits can be controlled by the settings of the needle valves, 15, in the respective lines. The circulating system is filled by means of the funnel, 7, and vent, 8. Drains for the



(a) Hevea Stock. (b) GR-S Stock.
Fig. 7.—X-ray Diffraction Patterns for Stretched Hevea and GR-S.

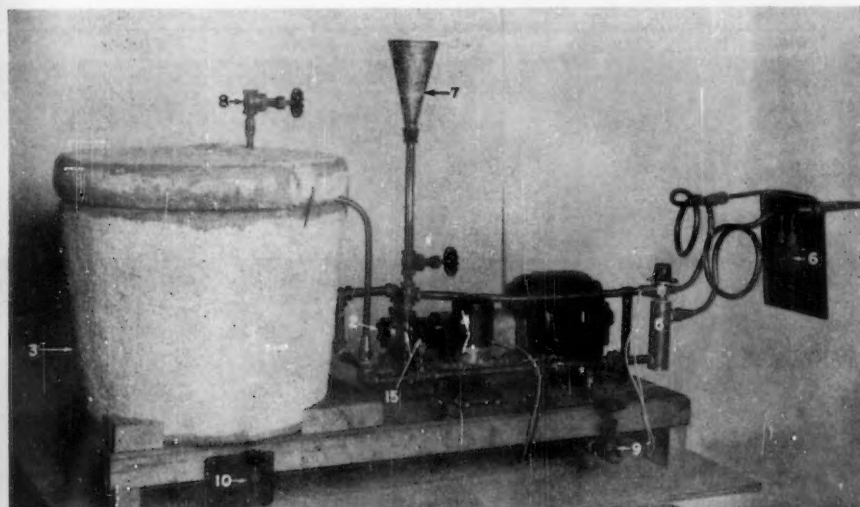


Fig. 8.—Cooling System for Low-Temperature X-ray Diffraction Work on Elastomers.

circulating system and cooling bath are provided for by valves 9 and 10, respectively.

The low-temperature camera is shown in Fig. 9. A $\frac{1}{4}$ -in. thick bakelite box 11, encloses the specimen holder, which, is mounted on the side closer to the X-ray tube, 13. The film cassette, 14, makes up one of the sides of the box. The side with the specimen holder is clamped to the collimating system, 12, and the remaining sides telescope on this side permitting adjustment of the specimen to film distance. Heat losses are minimized by the bakelite box and the fact that the circulating system and cooling chamber are covered with insulation.

The sample holder (Fig. 10) consists of two parts: a square copper block

with a U-channel through which the refrigerant is circulated and a circular copper disk which fits into a recess in the block. The disk and block have a common center hole to permit passage of the X-ray beam. The sample is placed in a groove in the disk. There is a tendency for ice crystals to form on the specimen due to condensation. This effect may be minimized by cementing of 0.001-in. cellophane windows coated with a glycerin-alcohol film on each side of the sample holder disk.

Transmission patterns were made on each of the stocks in the special low-temperature camera. Thin strips of the rubber samples were exposed in the unstretched state, both at room temperature (with no aging), and frozen at -70 F. after being aged in a constant

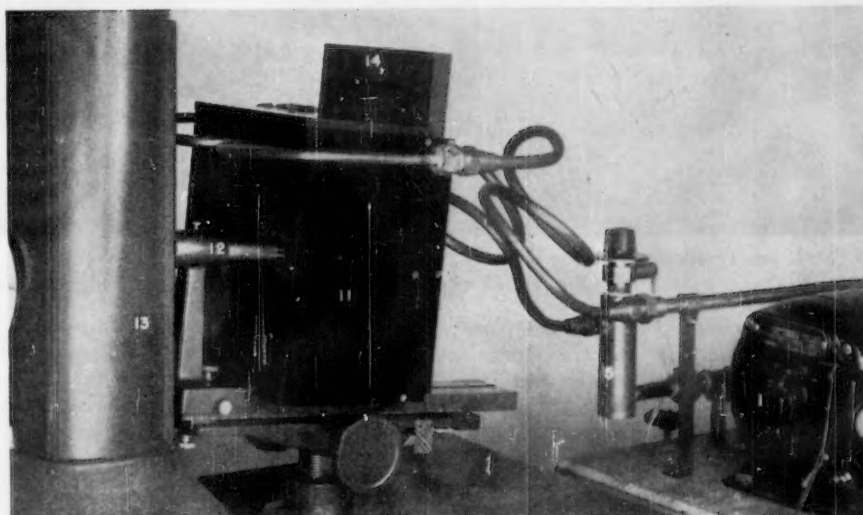


Fig. 9.—Camera for Low-Temperature X-ray Diffraction.

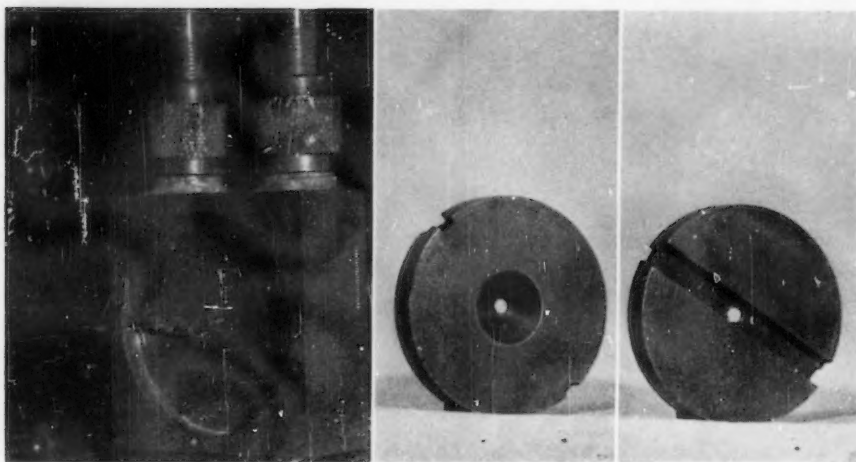
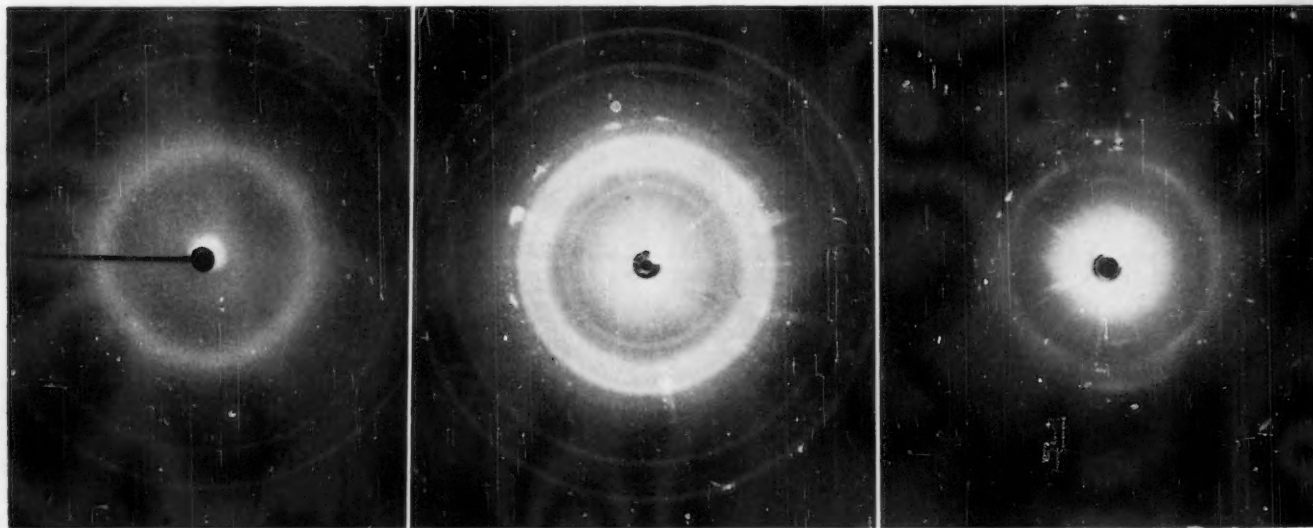


Fig. 10.—Sample Holder for Low-Temperature X-ray Diffraction.

TABLE II.—TYPE PATTERNS OBTAINED UNDER DIFFERENT EXPOSURE CONDITIONS FOR RUBBER STOCKS.

Conditions of Exposure	Hevea	GR-S	Nitrile Rubber	GR-M	Thiokol FA	GR-I
Room temperature, unstretched	Diffuse rings.	Diffuse rings.	Diffuse rings.	Diffuse rings.	Diffuse rings.	Diffuse rings.
Room temperature, stretched	Fiber pattern. 400 per cent Elong.	Diffuse rings. 400 per cent Elong.	Diffuse rings. 400 per cent Elong.	Fiber pattern. 400 per cent Elong.	Diffuse rings. 350 per cent Elong.	Fiber pattern. 300 per cent Elong.
Stretched at room temperature Frozen at -70 F.	Fiber pattern. 400 per cent Elong.	Diffuse rings. 400 per cent Elong.	Diffuse rings. 400 per cent Elong.	Fiber pattern. 400 per cent Elong.	Fiber pattern. 350 per cent Elong.	Fiber pattern. 300 per cent Elong.
No aging Stretched at -30 F. Frozen at -70 F.	Fiber pattern. 350 per cent Elong.	Diffuse rings. 400 per cent Elong.	Diffuse rings. 250 per cent Elong.	Fiber pattern. 360 per cent Elong.	Diffuse rings. 340 per cent Elong.	Fiber pattern. 300 per cent Elong.
Aged at -25 F. for 64 hr. Unstretched Frozen at -70 F.	Sharp rings.	Diffuse rings.	Diffuse rings.	Diffuse rings.	Diffuse rings.	Diffuse rings.
Aged at -25 F. for 64 hr. Stretched Frozen at -70 F.	Sharp rings and intense spots 225 per cent Elong.	Diffuse rings. 230 per cent Elong.	Diffuse rings. 240 per cent Elong.	Fiber pattern. 370 per cent Elong.	Fiber pattern. 200 per cent El.	Fiber pattern. 260 per cent Elong.



(a) Room Temperature, Unaged.

(b) Aged, Unstretched.

(c) Aged, Stretched.

Fig. 11.—X-ray Diffraction Patterns for Hevea Under Various Treatments.

temperature box. The aged samples were frozen on dry ice and then transferred to the camera in the sample holder, which was precooled on dry ice. This prevented the sample from warming up during transfer to the camera.

To obtain patterns for the aged, stretched samples, the following procedure was used. The aged samples

were immersed in an alcohol-dry ice bath at a temperature of -30 F., which is low enough to prevent "melting" of the rubber crystals yet high enough to permit elongation of the samples. After stretching the samples in the bath, the temperature was reduced to -70 F., to freeze the samples in the stretched condition. The stretched

frozen sample was placed in the groove in the sample holder disk, which was imbedded in a block of dry ice. The sample and disk were then quickly transferred to the camera. A technique similar to the one described above was used for the unaged stretched specimens.

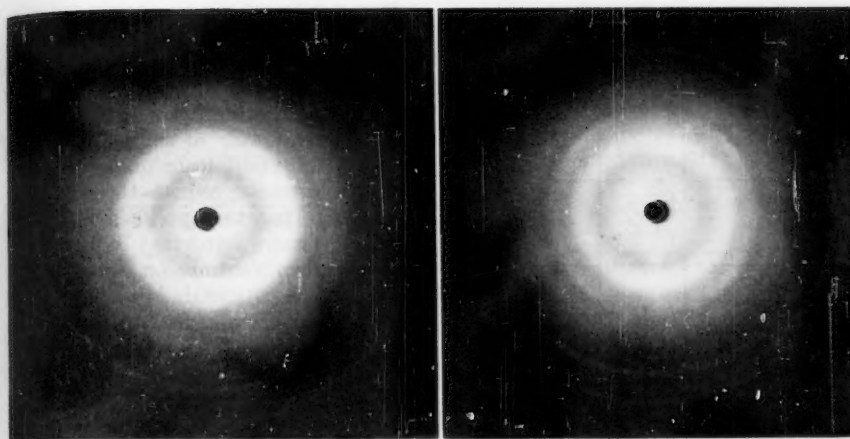
The elongation was determined by measuring the length of the frozen stretched sample and comparing it with the length of the relaxed sample measured after removal from the camera.

Patterns were taken on all samples under the following conditions:

- (a) room temperature unstretched,
- (b) room temperature stretched,
- (c) stretched at room temperature and frozen,

- (d) unaged, stretched at -30 F., frozen,
- (e) aged at -25 F. for 64 hr. unstretched, frozen and
- (f) aged at -25 F. for 64 hr., stretched, frozen.

A tabular summary of all results is contained in Table II. Samples of



(a) Aged, Unstretched.

(b) Aged, Stretched.

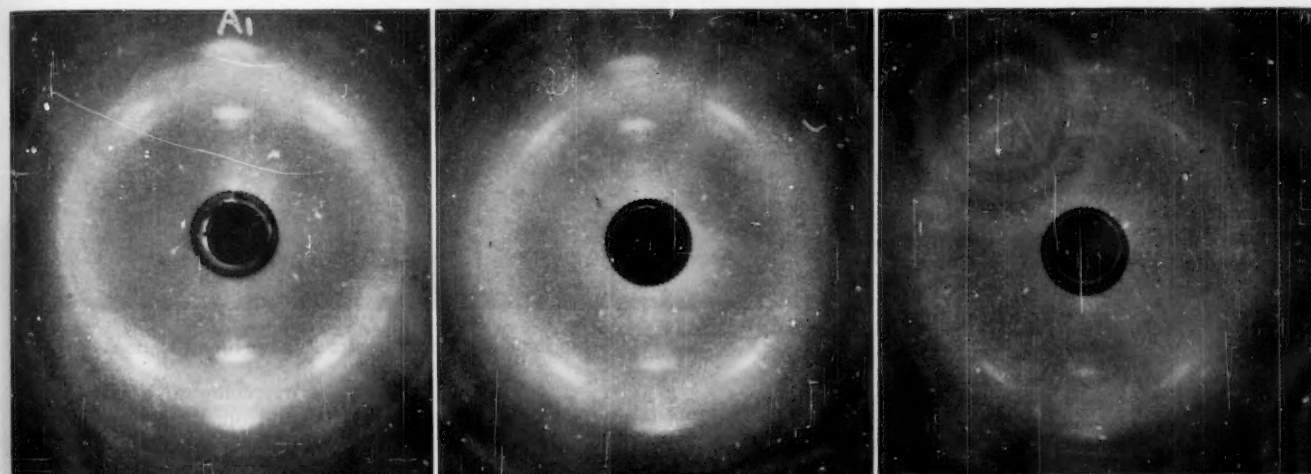
Fig. 12.—X-ray Diffraction Patterns of GR-I.

typical patterns of Hevea and GR-I are shown in Figs. 11 and 12. Examination of Table II shows that Hevea, GR-M, Thiokol FA, and GR-I can be made to produce characteristic crystalline patterns under one or more conditions reported, whereas GR-S and nitrile rubbers show diffuse rings under all conditions of test.

using X-ray diffraction methods for semiquantitative estimation of a constituent in a mixture which crystallizes upon stretching.

It must be noted, however, that such a graph can be applied only to mixtures prepared in an identical manner. Use of varying grades of reclaimed stocks may result in errors of measurement, in

view of the variable density of the fiber pattern spots. The reclaiming process appears to destroy the crystallite formation properties of Hevea rubber, upon stretching, by virtue of a breakdown in molecular size during mastication. The severity of the mastication process and the grade of reclaimed stock employed would determine the extent of this breakdown. Hence, in some instances a reclaimed stock may exhibit crystallite formation when stretched, with a consequent recording of a fiber pattern upon X-ray diffraction analysis. However, use of a graph such as this gives a measure of the relative amount of crystallized rubber present in the mixture, so that if the reclaimed rubber is of a grade which can be crystallized by stretching, the build-up of the pattern would signify that effectively the quantity of crude rubber present is greater. It must be understood that for measurements such as this, all experimental conditions should be standardized, including thickness of sample, exposure conditions, and especially the percentage elongation of the rubber sample. This latter factor is of particular impor-



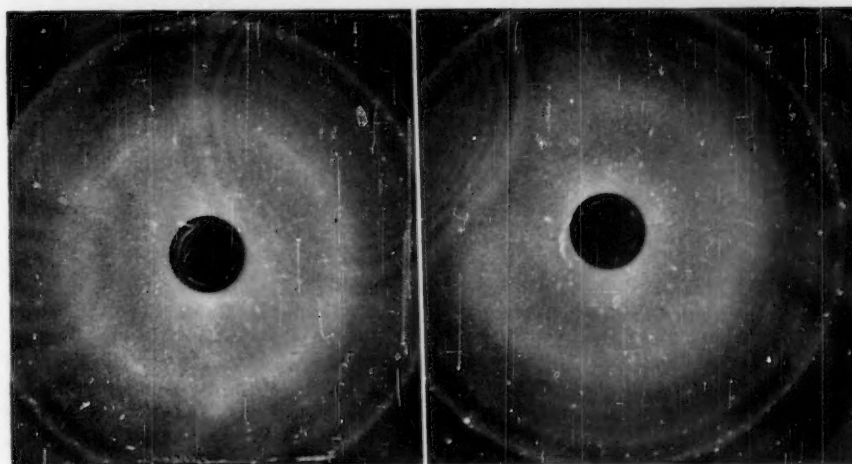
(a) 100 per cent Crude.

(b) 75 per cent Crude.

(c) 50 per cent Crude.

QUANTITATIVE ESTIMATION OF RUBBER MIXTURES BY FILM TECHNIQUES

To determine whether film techniques could be used for the estimation of rubber mixtures, samples of known content of crude and reclaimed stocks were run using samples stretched at room temperature as explained above with the setup of Fig. 6. The results of this test are shown in Fig. 13. A study of the patterns reveals that the intensity of interference spots due to crude rubber increases with increasing crude rubber content. The densities of A_1 spots, measured with an indicating type of photoelectric densitometer, plotted against the percentage of crude rubber yield a relationship shown in Fig. 14. This curve indicates the possibility of



(d) 25 per cent Crude.

(e) 0 per cent Crude.

Fig. 13.—X-ray Patterns for Mixtures of Crude and Reclaimed Rubber.

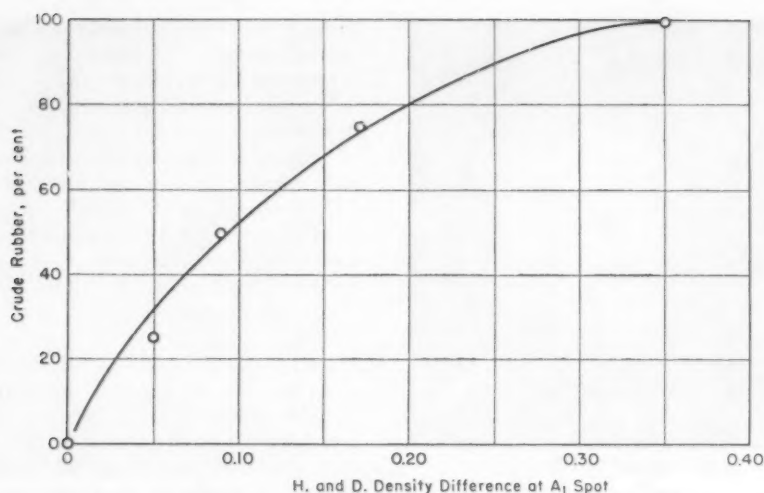


Fig. 14.—Density versus Per Cent Crude Rubber in Mixtures.

tance because, although the positions of the diffracted spots are characteristic of the rubber elastomer, the intensity of the spots depends upon the quantity of crystallites present, which is determined (among other things) by the percentage elongation of the sample.

GEIGER COUNTER SPECTROMETER TECHNIQUES FOR THE QUALITATIVE IDENTIFICATION OF RUBBER TYPES

During the course of this test an X-ray spectrometer became available for use in the laboratory. The X-ray spectrometer described by Friedman (15) differs from a conventional film X-ray diffraction unit in that individual elements of diffraction cones are recorded in succession by means of a Geiger counter which operates a recording potentiometer. Since utilization of this instrument would result in many

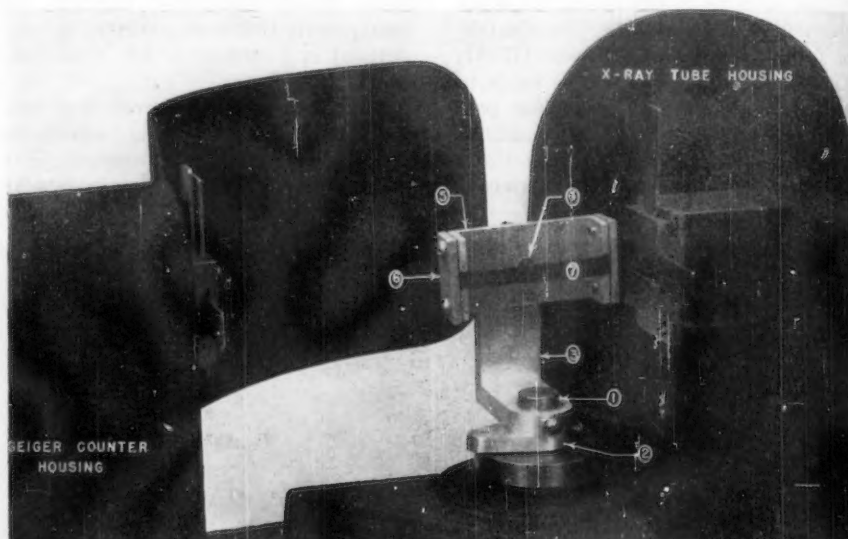


Fig. 15.—Test Setup for X-ray Spectrometer Rubber Identification.

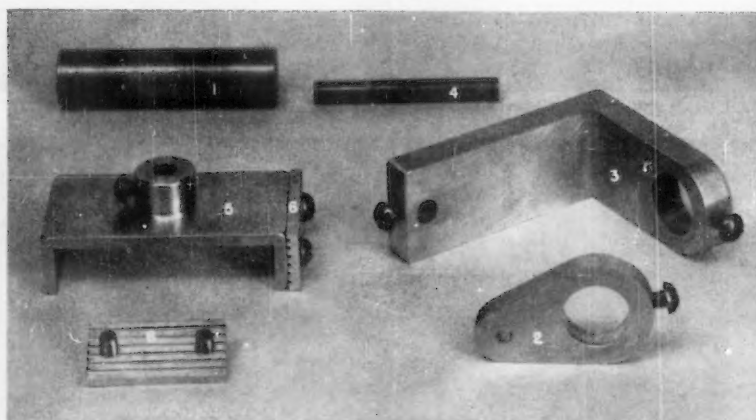


Fig. 16.—X-ray Spectrometer Sample Holder for Rubber.

advantages over film methods, it was decided to investigate the feasibility of applying the spectrometer to the study of rubbers. To begin with, since the diffraction effects produced by these rubbers occur below 20 deg. θ (Bragg angle), the time necessary to run

a spectrometer trace is of the order of 20 min. at 2 rpm. scanning speed, as compared with an average of 2 hr. for film exposures. The need for film processing and densitometry is eliminated, as well as the problem of determining specimen to film distance. The spec-

trimeter traces can be interpreted more easily, as both reflection angles and intensities can be taken directly from the trace. The use of larger specimens makes for better sampling and minimizes the danger of sample breakage.

The reflection technique utilized in the spectrometer reduces the difficulties of controlling the specimen thickness, which is more important in film techniques when transmission is used. The intensity variations due to differences in sample thickness in film techniques are far greater than the intensity variations due to penetration and absorption effects of the incident radiation in the spectrometer. The difficulties due to high background intensities which are inherently characteristic of film patterns in regions where rubber reflections usually

occur are overcome to a large extent when the spectrometer is used. This is due to the fact that the sensitivity of the silver halides to these radiations is much higher than that of the Geiger counter. As a result, the important reflections of rubber samples are more distinct in spectrometer traces than in film patterns where they are obscured by high background effects. One of the disadvantages associated with the use of large samples is that elongations commensurate with those obtained on thin samples cannot be obtained readily. Another disadvantage of the spectrometer method is the difficulty of getting quantitative reproducibility of the traces due in part to errors inherent in the instrument, particularly when using the recorder at fast scanning rates. A scanning rate of 2 rpm. was selected to reduce the width of the diffraction maxima, and because only qualitative differences were investigated in this report. The faster scanning rate also

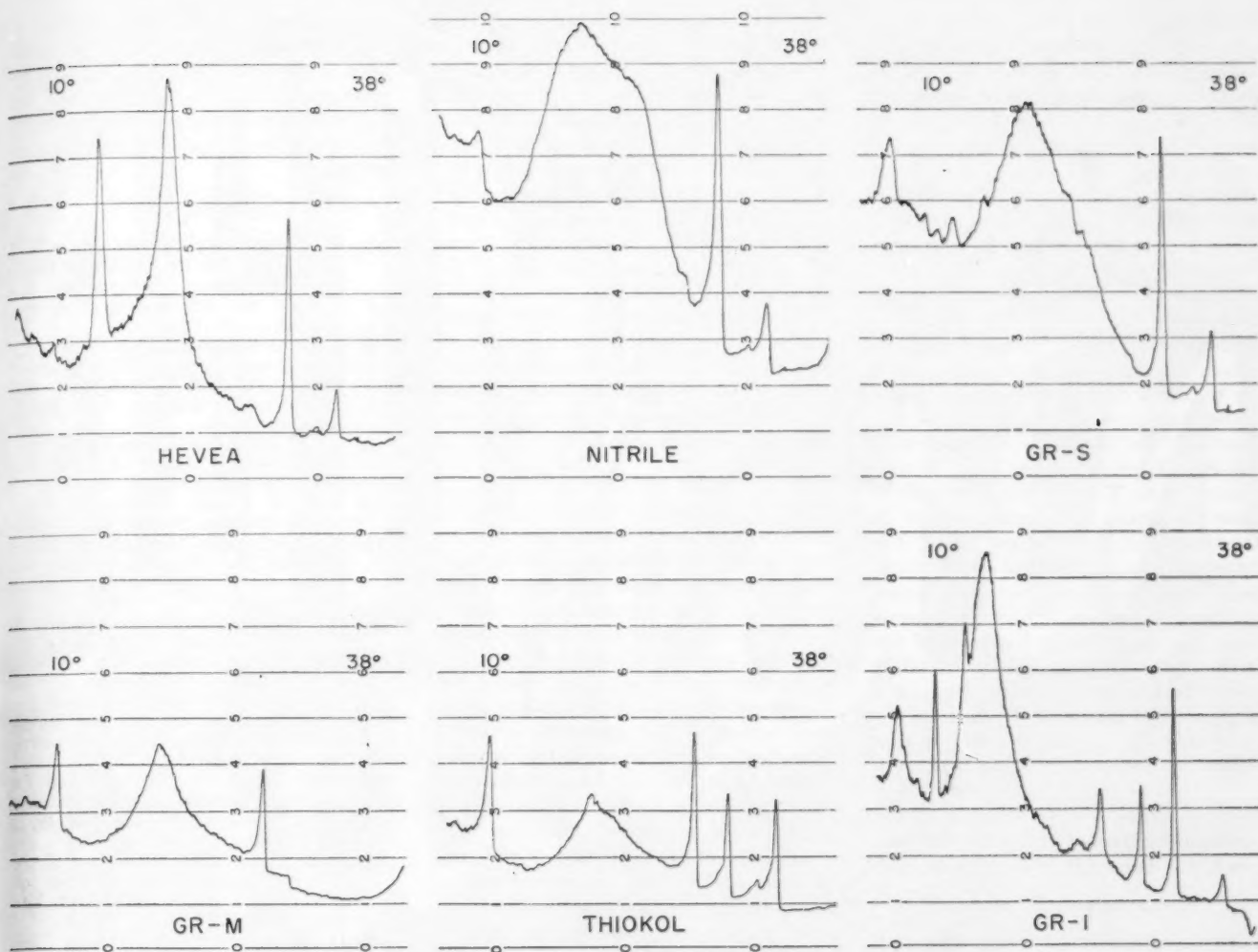


Fig. 17.—X-ray Spectrometer Patterns for Six Representative Rubber Types.

reduced the time necessary to run a pattern.

A North American Phillips Geiger counter X-ray spectrometer with Brown recorder and nickel-filtered copper radiation was used in this work. A special sample holder, built in the laboratory, for holding the stretched rubber samples is shown in Figs. 15 and 16. In these photographs, 1 is the specimen holder shaft, 2 is the positioning collar, 3 is an L-shaped bracket which holds shaft 4 on which a channel-shaped piece, 5, is mounted equipped with clamps, 6, for holding the rubber specimen, 7. The relationships of the sample holder to the X-ray tube and Geiger counter are shown in Fig. 15 while Fig. 16 shows details of sample holder parts.

Samples about $\frac{1}{2}$ in. wide and $\frac{1}{8}$ in. thick were stretched as much as possible, clamped in the sample holder, and placed in the spectrometer as shown in Fig. 15. The specimen was set with the direction of stretching horizontal, as this position yielded the highest intensities, and was aligned so that it was tangent to the focusing circle of the instrument.

This was accomplished by the use of a rod drilled out at the bottom to fit over the specimen holder shaft, and machined flat along a diameter at the top.

Traces were run at a scanning rate of 2 rpm. from 38 deg. (2θ) to about 4 deg. (2θ), which range included the important diffraction lines of the samples being studied. All traces were made with the same amplitude and damping settings except for GR-I, in which case it was necessary to decrease the amplitude setting. Figure 17 shows the traces for the standard stocks of the six rubber types in the stretched condition. It should be noted that the angles indicated on the traces are in degrees 2θ . Table III shows a comparison of the characteristic reflections of the rubbers (the

compounding ingredients' reflections are not included). The d values were measured at the points where the maxima occurred. Since most of the stocks contain zinc oxide, its reflections were used as an internal standard to overcome errors due to slippage and non-uniformity of the recorder chart paper.

The patterns for all stocks in the stretched condition except for nitrile rubber and GR-S showed an improvement in intensity and sharpness of the diffraction maxima over those obtained in the unstretched state, as was evident from the film work. In the case of Hevea, the single diffuse ring gives rise to two sharp reflections in the stretched sample. This occurs because the spectrometer pattern represents a trace of

TABLE III.—X-RAY SPECTROMETER DATA FOR REPRESENTATIVE RUBBER STOCKS.

Rubber Stock	Elongation, per cent	d Values for Characteristic Maxima, Å	Width of Reflection, 2θ , deg.	Intensity over Background	Shape of Reflection
Hevea.....	430	4.27	8.0	6.0	Symmetrical
		6.22	2.0	4.39	Symmetrical
GR-M.....	300	4.57	11.2	2.20	Symmetrical
Nitrile.....	330	4.69	16.2	4.99	Unsymmetrical
Thiokol FA.....	100	4.57	12.4	1.72	Unsymmetrical
GR-S.....	220	4.48	16.4	4.27	Symmetrical
GR-I.....	350	5.99	10.4	5.71	Symmetrical

the intensities through one plane of the film diffraction pattern. Thus, the two maxima correspond to the intense spots at 12 or 6 o'clock shown in the stretched Hevea film pattern, Fig. 7. A similar analogy holds true for the traces of the other stocks.

Consideration of traces, Fig. 17, and the d values of Table III indicates that Hevea and GR-I stocks can be readily identified, since the d values of their characteristic maxima are quite distinct from those of the other four stocks, as well as each other. Thiokol, GR-M, GR-S, and nitrile rubber patterns cannot be resolved on the basis of d values alone, since these are very close to one another. On the basis of intensities, GR-S and nitrile rubber may be separated from Thiokol FA and GR-M since the former exhibit much higher intensities over background than the latter two, and these values are comparable, as the traces were made under identical conditions. Further separation may be made on the basis of the shape of the diffraction maxima, GR-S and GR-M being symmetrical in shape while nitrile rubber and Thiokol are unsymmetrical. Another factor which might be utilized in resolving these stocks is the width of the diffraction maxima, those for GR-S and nitrile rubber being broader than Thiokol and GR-I, see Table III.

SUMMARY OF RESULTS

The results of film work to date may be summarized as follows. At room temperature, Hevea, GR-M, and GR-I will produce fiber patterns on stretching, and these patterns can be readily differentiated from one another. Aging and freezing of Hevea stock produces a pattern containing sharp diffraction rings similar to the type produced by randomly oriented crystalline materials. In the case of Thiokol FA, a fiber pattern is obtained by either aging, stretching, and freezing or stretching at room temperature and freezing. While the fiber patterns obtained for Thiokol FA and GR-M are similar, they may be differentiated by considering the conditions necessary to produce them. GR-S and nitrile rubbers did not yield characteristic patterns under any of the conditions of treatment.

The results obtained on the X-ray spectrometer indicate that the six stocks considered in this test can be made to yield distinctive patterns when examined in the stretched condition. This is accomplished through consideration of the location, intensity, breadth, and shape of the characteristic reflections of the different stocks. Traces run on the six cold-resistant stocks showed the characteristic rubber reflections similar to the standard types.

Results on the semiquantitative estimation of constituents in mixtures of crude and reclaimed rubbers indicate possibilities of extending X-ray diffraction applications to mixtures of other representative rubber types.

FUTURE WORK

In the immediate future, methods for extending the use of the X-ray spectrometer to the identification of compounding ingredients will be developed. This work appears to be justified since potential economies over film techniques in time and money are indicated.

Further problems which could be investigated by X-ray diffraction methods include: (1) quantitative determination of mixtures of the several rubber types with a view of setting up working curves to be utilized in the analysis of unknown mixtures, (2) establishment of combined X-ray diffraction and optical emission spectroscopy techniques for the quantitative analysis of compounding ingredients, and (3) determination of crystallinity induced as a function of elongation and relaxation under various conditions for representative rubber stocks and mixtures.

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